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U.S. DEPARTMENT OF COMMERCE PATENT AND TRADEMARK OFFICE

ATTORNEY'S DOCKET NUMBER

TRANSMITTAL LETTER TO THE UNITED STATES
DESIGNATED/ELECTED OFFICE (DO/EO/US)
CONCERNING A FILING UNDER 35 U.S.C. 371

2651-0028-2X PCT

U.S. APPLICATION NO. (IF KNOWN, SEE 37 CFR

09/380630

INTERNATIONAL APPLICATION NO.
PCT/JP98/01110INTERNATIONAL FILING DATE
17 March 1998PRIORITY DATE CLAIMED
21 March 1997

TITLE OF INVENTION

METHOD FOR MARKING MATERIALS AND MATERIALS USED FOR MARKING

APPLICANT(S) FOR DO/EO/US

Hirotooshi HAYAKAWA, et al

Applicant herewith submits to the United States Designated/Elected Office (DO/EO/US) the following items and other information:

1. ☒ This is a **FIRST** submission of items concerning a filing under 35 U.S.C. 371.
2. ☐ This is a **SECOND** or **SUBSEQUENT** submission of items concerning a filing under 35 U.S.C. 371.
3. ☒ This is an express request to begin national examination procedures (35 U.S.C. 371(f)) at any time rather than delay examination until the expiration of the applicable time limit set in 35 U.S.C. 371(b) and PCT Articles 22 and 39(1).
4. ☒ A proper Demand for International Preliminary Examination was made by the 19th month from the earliest claimed priority date.
5. ☒ A copy of the International Application as filed (35 U.S.C. 371 (c) (2))
 - a. ☐ is transmitted herewith (required only if not transmitted by the International Bureau).
 - b. ☒ has been transmitted by the International Bureau.
 - c. ☐ is not required, as the application was filed in the United States Receiving Office (RO/US).
6. ☒ A translation of the International Application into English (35 U.S.C. 371(c)(2)).
7. ☒ A copy of the International Search Report (PCT/ISA/210).
8. ☒ Amendments to the claims of the International Application under PCT Article 19 (35 U.S.C. 371 (c)(3))
 - a. ☐ are transmitted herewith (required only if not transmitted by the International Bureau).
 - b. ☐ have been transmitted by the International Bureau.
 - c. ☐ have not been made; however, the time limit for making such amendments has NOT expired.
 - d. ☒ have not been made and will not be made.
9. ☐ A translation of the amendments to the claims under PCT Article 19 (35 U.S.C. 371(c)(3)).
10. ☒ An oath or declaration of the inventor(s) (35 U.S.C. 371 (c)(4)).
11. ☐ A copy of the International Preliminary Examination Report (PCT/IPEA/409).
12. ☐ A translation of the annexes to the International Preliminary Examination Report under PCT Article 36 (35 U.S.C. 371 (c)(5)).

Items 13 to 18 below concern document(s) or information included:

13. ☐ An Information Disclosure Statement under 37 CFR 1.97 and 1.98.
14. ☐ An assignment document for recording. A separate cover sheet in compliance with 37 CFR 3.28 and 3.31 is included.
15. ☐ A **FIRST** preliminary amendment.
A **SECOND** or **SUBSEQUENT** preliminary amendment.
16. ☐ A substitute specification.
17. ☐ A change of power of attorney and/or address letter.
18. ☐ Certificate of Mailing by Express Mail
19. ☒ Other items or information:

Notice of Priority

PCT/IB/304

PCT/IB/308

Drawings (15 Sheets)

Request For Consideration of Documents Cited in International Search Report

U.S. APPLICATION NO. (IF KNOWN, SEE 37 CFR 09/380630)	INTERNATIONAL APPLICATION NO. PCT/JP98/01110	ATTORNEY'S DOCKET NUMBER 2651-0028-2X PCT
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20. The following fees are submitted:

BASIC NATIONAL FEE (37 CFR 1.492 (a) (1) - (5)) :

- | | |
|--|-----------------|
| <input checked="" type="checkbox"/> Search Report has been prepared by the EPO or JPO | \$840.00 |
| <input type="checkbox"/> International preliminary examination fee paid to USPTO (37 CFR 1.482) | \$670.00 |
| <input type="checkbox"/> No international preliminary examination fee paid to USPTO (37 CFR 1.482) but international search fee paid to USPTO (37 CFR 1.445(a)(2)) | \$760.00 |
| <input type="checkbox"/> Neither international preliminary examination fee (37 CFR 1.482) nor international search fee (37 CFR 1.445(a)(2)) paid to USPTO | \$970.00 |
| <input type="checkbox"/> International preliminary examination fee paid to USPTO (37 CFR 1.482) and all claims satisfied provisions of PCT Article 33(2)-(4) | \$96.00 |

ENTER APPROPRIATE BASIC FEE AMOUNT =**\$840.00**
 Surcharge of **\$130.00** for furnishing the oath or declaration later than ☐ 20 ☐ 30 months from the earliest claimed priority date (37 CFR 1.492 (e)).
\$0.00

CLAIMS	NUMBER FILED	NUMBER EXTRA	RATE
Total claims	31 - 20 =	11	x \$18.00
Independent claims	3 - 3 =	0	x \$78.00

\$198.00**\$0.00**

Multiple Dependent Claims (check if applicable).

☒**\$260.00****TOTAL OF ABOVE CALCULATIONS =****\$1,298.00**

Reduction of 1/2 for filing by small entity, if applicable. Verified Small Entity Statement must also be filed (Note 37 CFR 1.9, 1.27, 1.28) (check if applicable).

☐**\$0.00****SUBTOTAL =****\$1,298.00**Processing fee of **\$130.00** for furnishing the English translation later than ☐ 20 ☐ 30 months from the earliest claimed priority date (37 CFR 1.492 (f)).☐**\$0.00****TOTAL NATIONAL FEE =****\$1,298.00**

Fee for recording the enclosed assignment (37 CFR 1.21(h)). The assignment must be accompanied by an appropriate cover sheet (37 CFR 3.28, 3.31) (check if applicable).

☐**\$0.00****TOTAL FEES ENCLOSED =****\$1,298.00**

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- ☒ A check in the amount of **\$1,298.00** to cover the above fees is enclosed.
- ☐ Please charge my Deposit Account No. _____ in the amount of _____ to cover the above fees.
A duplicate copy of this sheet is enclosed.
- ☒ The Commissioner is hereby authorized to charge any fees which may be required, or credit any overpayment to Deposit Account No. **15-0030** A duplicate copy of this sheet is enclosed.

NOTE: Where an appropriate time limit under 37 CFR 1.494 or 1.495 has not been met, a petition to revive (37 CFR 1.137(a) or (b)) must be filed and granted to restore the application to pending status.

SEND ALL CORRESPONDENCE TO:

OBLON, SPIVAK, McCLELLAND, MAIER & NEUSTADT, P.C.
 Crystal Square Five, Fourth Floor
 1755 Jefferson Davis Highway
 Arlington, Virginia 22202
 US

Telephone Number (703) 414-7000
 Facsimile Number (703) 414-7770
 REGISTRATION NUMBER 30,996

SIGNATURE

Marvin J. Spivak

NAME

24,913

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DESCRIPTION

METHOD FOR MARKING MATERIALS AND

MATERIALS USED FOR MARKING

Technical Field

The present invention relates to a method and materials for forming a mark such as a product number or a lot number on glass or on a laser transmittive body used as a component of a liquid crystal panel, a plasma display panel or a semiconductor substrate or the like.

Background Art

Methods of marking on a transparent body such as glass or a laser transmittive body have been disclosed in Japanese Patent Laid-open No. 008634/1994 and Japanese Patent Laid-open No. 155920/1994.

Fig. 16 and Fig. 17 show the prior art disclosed in Japanese Patent Laid-open No. 008634/1994. In Fig. 16, the numeral 31 represents a glass plate, 32 a metal plate made of material such as stainless steel, phosphor bronze or aluminum, 33 a condensing lens, and 34a and 34b are x-axis and y-axis rotation mirrors for scanning the laser beam LA, and 35a and 35b are galvanometer scanners of x-axis and y-axis rotation mirrors, respectively. According to the marking method of the prior art, the glass plate 31

and the metal plate 32 are closely contacted with each other, a laser beam is applied through the glass to the surface of metal plate 32, a desired pattern such as of a character, a diagram or a symbol, is formed with the heated metal of metal plate 32 by the laser beam irradiation is deposited to the glass plate. By applying the laser beam LA as shown in Fig. 17(a) and separating glass plate 31 from metal plate 32 as shown in Fig. 17(b), metal particles MR deposited to the surface of glass plate 31a is obtained showing the marking of the pattern having the metal color of metal plate 32.

Fig. 18 shows the prior art disclosed in Japanese Patent Laid-open No. 155920/1994. This conventional example uses the same apparatus as shown in Fig. 16, and as shown in Fig. 18, chips 36a and 36b of a thickness of 0.2 to 0.5 mm are placed between glass plate 31 and metal plate 32 to form a gap therebetween. Then a laser beam is applied with a desired pattern such as of a character, a diagram or a symbol through the glass plate to the surface of metal plate 32 to locally heat the surface of metal plate 32a irradiated by the laser beam, thereby emitting matter such as metal particles MR, ions or plasma from metal plate 32 in the direction of the arrow A. As shown in Fig. 18, a part of the matter emitted strikes the surface of confronting glass plate 31, thereby removing

surface 31a of glass plate 31 by the shock while forming dent 31b. When glass plate 31 is separated from metal plate 32, dent 31b appears to be white or milky white. Therefore, when the laser beam is applied with a desired pattern while scanning, white marks are formed showing the pattern on the surface of glass plate 31. As described above, there have been two methods for marking on a transparent body such as a glass plate.

However, in the prior art disclosed in Japanese Patent Laid-open No. 008634/1994, since the evaporation area of the metal spreads wider than the diameter of the laser beam, when the laser beam is applied to the metal, it is unable to form minute marks. Further, when a laser beam is applied to metal, since the temperature of the metal is raised as high as the fusion temperature and the metal is closely contacted with the glass surface, the temperature of the closely contacted glass plate is also highly raised locally. Consequently, a crack is occasionally generated in the glass plate which is weaker than the metal plate in thermal shock, or at the worst, the glass plate is broken.

On the other hand, in the prior art disclosed in Japanese Patent Laid-open No. 155920/1994, there is a gap of 0.2 to 0.5 mm between the glass plate to be marked and the metal plate, and since matter such as metal particles

MR, ions or plasma generated by irradiation of the laser beam and spread into a two dimensional area, it is not possible been unable to form delicate marking. Further, since the mark is white or milky white in color, it is hard to recognize visually, and with an instrument having a photodetector such as a bar-code reader, it is completely impossible to recognize the marked codes or characters. Further, since a dent is formed in the surface of the glass plate, the bending strength of the glass decreases remarkably. Particularly, in the case of thin glass plate of the thickness of 1 mm or less used as liquid crystal panels, the glass plate is sometimes broken by the bending stress due to dead weight, while precludes mechanical transportation.

Recently, forming marks on a liquid crystal panel or a plasma display panel has been not only for forming product numbers, but also for forming QR Code, Data Code, Veri Code or two-dimensional bar codes or bar codes on a glass plate in the initial process of manufacture for process control of each panel substrate. Therefore, it is greatly required that the mark is of easy recognition, uniform quality, stable and unchanging in quality during manufacture, and low cost .

However, with a marking material of the prior art, the mark such as symbol etc. disappears due to heat

treatment in the manufacturing process, and it is difficult to form a predetermined configuration in some marking material, both of which result in increased costs.

Also, in the methods of the prior art, there are problems such as unevenness of thickness of the film on which the mark is formed on a glass plate, part of a mark disappears, and at worst the code can not be read with a data code reader. This is because the film of the code mark is required to shield light completely for reading a code with a code reader, but if there is irregularity in film thickness, the quantity of light shielded by the film of the mark varies, and correct reading of the mark is interrupted thereby.

Disclosure of Invention

It is an object of the present invention to provide a method of marking for easily and uniformly forming a minute mark, which has not been realized by the prior art, on a transparent body such as glass or a laser transmittive body. Such a marking method is required to obtain easy visual recognition and instrument reading of a mark, while supplying a marked plate with high strength against bending and which does not cause to crack or fracture from the thermal shock given to the glass plate.

The present invention also provides a marking

material for a heat-and-chemicals-proof code mark.

The present invention achieves the above stated object by providing a method for marking materials using a material to be marked and a marking material consisting of a transparent body or a laser transmittive body, comprising a first process of placing the surface of said material to be marked and the surface of said marking material together with a desired gap therebetween, vaporizing said marking material by irradiating it through said material to be marked with a beam of a first laser power while scanning with the laser beam, and depositing a deposit vaporized from said marking material to a predetermined portion of said material to be marked and

a second process of removing or denaturalizing said deposit deposited to the surface of said material to be marked with a laser beam of a second laser power, whereby, patterns of characters, diagrams or symbols are formed on said material to be marked.

The present invention further provides a method for marking materials to form patterns of characters, diagrams or symbols on a material to be marked consisting of a transparent body or a laser transmittive body comprising the steps of creating a desired gap between the surface of said material to be marked and the marking material,

reacting the gas which exists in said gap from the

vaporization of said marking material irradiated by said laser beam with the vaporized marking material, and adhering the reaction product onto the surface of said material to be marked.

In addition, the present invention provides a method for marking materials comprising steps of

forming patterns of characters, diagrams or symbols by changing the light transmittivity or the light reflection factor of the deposit consisting of the marking material deposited to the transparent body or laser transmittive body by irradiating with a laser beam.

A metal, an alloy, an intermetallic compound or a metal compound, or a compound which includes at least one of said metal, alloy, intermetallic compound or metal compound, and in particular a steel or stainless steel is used as the marking material.

According to the present invention, since marking is done by adhering a film onto a glass plate in a first process and partially removing the film in a second process, clear fine marks can be obtained effectively. Also, since the glass plate is not damaged, the glass plate does not break and moreover, a mark with a high bending strength can be effectively provided.

Since the present invention employs stainless steel or steel as a marking material, a heat-and-chemicals-proof

code mark can be formed, thereby assuring reliable code recognition on the production line.

Also, as thin film is used on the marking material comprising of a laser transmittive body like a glass plate, a code mark can be formed having a uniform film thickness, making stable code recognition possible.

Brief Description of Drawings

Fig. 1 is a sectional view and a plan view of a marking process with reference to embodiment 1.

Fig. 2 is a time dependence of YAG laser power.

Fig. 3 is a variation of YAG laser power.

Fig. 4 is a sectional view and a plan view of the marking process with reference to embodiment 2.

Fig. 5 is a sectional view and a plan view of the marking process with reference to embodiment 3.

Fig. 6 is a plan view of Two-dimensional Code.

Fig. 7 is a plan view showing laser scanning in the first process.

Fig. 8 is a plan view showing laser scanning in a cell of Two-dimensional Code.

Fig. 9 is a plan view showing laser scanning of the character L pattern.

Fig. 10 is a view showing the relationship between the thickness of a film manufactured in the first process

and the laser power with reference to embodiment 5.

Fig. 11 is a sectional view and a plan view of the marking process with reference to embodiment 6.

Fig. 12 is a sectional view and a plan view of the marking process with reference to embodiment 7.

Fig. 13 is a sectional view and a perspective view of the marking process with reference to embodiment 8.

Fig. 14 is a view showing the surface configuration of a mark manufactured by the marking method of embodiment 8.

Fig. 15 is a type view of the marking apparatus used in the marking method of embodiment 10.

Fig. 16 is a perspective view showing the structure of the main parts of a marking apparatus of the prior art.

Fig. 17 is a sectional view of the prior art making.

Fig. 18 is a sectional view showing the appearance of marking in the prior art.

Best Mode for Carrying Out the Invention

Embodiments of the present invention will be described below with reference to the drawings.

(Embodiment 1)

Fig. 1 is a sectional view and a plan view showing the process of the marking method of embodiment 1. In the process, chips 6a and 6b of 100 μ m in thickness are

placed on metal plate 1 formed from titanium as the marking material, and glass plate 2, the material to be marked, is placed on the chips to form a gap of $100\text{ }\mu\text{m}$ between metal plate 1 and glass plate 2 (Fig. 1(a)). By using a YAG laser device and adjusting the focus of laser beam LA of $40\text{ }\mu\text{m}$ in beam diameter onto metal plate 1, a laser beam to a desired area (2 mm square) scanning at $50\text{ }\mu\text{m}$ interval by means of first laser power (in the present embodiment, not less than 30 W or 30 mJ/pulse).

Accordingly, the metal of metal plate 1 irradiated by the laser beam LA is heated and melted, partially evaporated, ionized or turned to plasma. The metal evaporated, ionized or turned to plasma chemically reacts with the gas in the gap between metal plate 1 and glass plate 2 to produce titanium monoxide, dititanium trioxide and then they deposite to surface 2a of glass plate 2. Through the first process described above, a black film M of $0.2\text{ }\mu\text{m}$ in thickness is formed on glass plate 2 in an area of 2 mm square (Fig. 1(b)).

Next, the second process is described. In Fig. 1(c), glass plate 2 formed in the first process is separated from marking material 1 in a stage not shown. By applying a laser beam of $40\text{ }\mu\text{m}$ diameter to film M on surface 2a of the separated glass plate by means of a second laser power (in the present embodiment, less than 30 W or 30 mJ/pulse),

the laser beam is scanned so as to form a predetermined character, diagram or symbol. Upon receiving the irradiation of the laser beam, the laser irradiated portion of the film M comprising titanium monoxide, dititanium trioxide are removed from surface 2a of glass plate 2. Thus, characters, diagrams or symbols 3 of a clear contrast are marked on glass plate 2. In the present embodiment, a two-dimensional Data Code mark M of 7 cells \times 7 cells is produced.

The beam diameter of the laser beam used in the present embodiment is 40 μ m as described above, and it succeeded in the embodiment in drawing a diagram having the resolution of the beam diameter. It is noted that when marking is performed according to the prior art using a laser beam of 40 μ m beam diameter, the resolution of the diagram is 50 μ m or more. In other words, according to the present embodiment, it is possible to form a minute mark having resolution the same as that of the laser beam diameter. Further, in the first process and the second process, a beam diameter of a different size can be employed and the laser power can also be changed for each process.

According to the present embodiment, a minute mark can be formed, and besides, since glass plate 2 and metal plate 1 are placed slightly separated from each other,

even if the metal is heated to a temperature as high as near the fusion temperature being irradiated by the laser beam, glass plate 2 does not crack or break. Also there is no risk of forming a hollow or a dent in the glass plate, the bending strength of the glass plate is not lowered thereby facilitating mechanical transportation. In the present embodiment, a YAG laser is used, but it is apparent that the same effect can be obtained with an excimer laser or a carbon dioxide gas laser if the laser beam can transmit through the material to be marked.

Further, titanium is used for the metal plate in the above embodiment, marking can be applied to the glass plate in the same way as in the present embodiment by using titanium-niobium alloy, titanium-zirconium alloy, titanium monoxide, titanium hydride, titanium-titanium monoxide compound or the like all including the titanium atom. Further, other than titanium, gold, iron, tungsten, molybdenum, lead, tin, silver, cobalt, chromium, copper, manganese, niobium, nickel, palladium, platinum, ruthenium, vanadium, Ni-Co, Cu-Ni, Nb_3Sn , Fe_3O_4 , $CrO-CO_2O_3$, Fe_2O_3-MgO can be used.

Further, by appropriately selecting the concentration of the gas contained in the gap, it becomes possible to make the same material as the metal plate deposit to glass plate without going through reaction

with the gas. Further even though a glass plate is used in the present embodiment, it is apparent that the same effect will be obtained by using a material with laser transmittive.

Further, in the present embodiment, particularly as the marking material, SUS301, SUS304, SUS310S, SUS405, SUS430, SUS447J1, SUS403, SUS410, SUS440A, S10C, S25C, S35C, S45C are used and marking is applied to the glass plate according to the procedure shown in Fig. 1. Fused silica is used as the glass plate. A two-dimensional Data Code thus marked on the glass plate has a clear contrast independent of the marking material used, and gives good recognition results by means of a data code reader at the time immediately after marking.

In a manufacturing process of the plasma display panel, the glass plate marked with a code is heat-treated in the manufacturing process at about 600 °C. In a manufacturing process of the liquid crystal panel, the glass plate marked with a code is heat-treated in manufacturing process at about 300 °C. In the present embodiment, the two-dimensional Data Code mark formed by using each of the above marking material was heat-treated in the atmosphere at a temperature of 650 °C for 30 minutes, and then subjected to the reading test. The reading test consists of reading of each two-dimensional Data Code for

10 seconds with 200 times reading and measures the number of correct reading of the code contents.

Table 1 shows the reading results picked after marking, the reading results picked after heat-treatment of 650 °C, 30 minutes in the atmosphere, and composition of marking materials.

[Table 1]

marking material	correct reading rates (%)		marking material composition (%)			
	after making	after heat-treatment in atmosphere 650°C, 30 min.	Fe	Cr	Ni	C
SUS301	100	85	72.9	17.0	7.0	0.15
SUS304	100	82	68.7	19.0	9.3	0.08
SUS304L	100	76	66.9	19.0	11.0	0.08
SUS301S	100	60	50.9	25.0	20.5	0.08
SUS405	100	93	85.7	12.3	0	0.08
SUS430	100	89	81.1	17.0	0	0.12
SUS447J1	100	78	68.9	30.3	0	0.01
SUS403	100	94	85.9	12.0	0.5	0.15
SUS410	100	92	84.3	13.0	0.5	0.2
SUS440A	100	52	79.9	17.0	0.5	0.65
S10C	100	94	99.2	0	0	0.1
S25C	100	92	99.08	0	0	0.22
S35C	100	70	98.65	0	0	0.35
S45C	100	50	98.55	0	0	0.45

It is noted that reading results are represented as correct reading rates. Further, it is also noted that a sum composition of each marking material does not reach 100 % in table 1, but this is because the marking material includes other material, such as Si, Mn not listed in the table. The correct reading rate just after marking is 100 % for every marking material.

In the present embodiment, when stainless steel is used as the marking material, the marking color changes a little after heat-treatment in the atmosphere at a temperature of 650 °C for 30 minutes, but the marked code further remains sufficiently recognizable for use. In case the marking material is stainless steel of an austenite group such as SUS301, SUS304, SUS310S, the correct reading rate of the code after heat-treatment is 60 to 85 %, and the more the marking material contains Ni and Cr components and the lesser contains Fe components, the worse the correct reading rate become. In the case the marking material is stainless steel of a ferrite group such as SUS405, SUS430, SUS447J1, the correct reading rate of the code after heat-treatment is 87 to 93 %, and the more the marking material contains Cr components and the lesser contains Fe components, the correct reading rate becomes worse a little. In the case the marking material is stainless steel of a martensite group such as SUS403, SUS410, SUS440, the correct reading rate of the code after heat-treatment is 52 to 94 %, and the more the marking material contains Cr components and the lesser contains Fe components, the correct reading rate becomes worse a little. However, the correct reading rate of SUS440A containing much carbon is 52 % and lower than that of other stainless marking material. On the one hand, any

one of steel group material S10C, S25C, S35C, S45C is used as the marking material, the correct reading rate of the code after heat-treatment is 50 to 94 %, and the more the marking material contains C components, the worse the correct reading rate becomes a little. When the carbon content is 0.25 % or less, the correct reading rate of the code after heat-treatment is 90 %, representing a high recognition ratio. From the above results, with an increased amount of Ni contents in the marking material, the correct reading rate after heat-treatment becomes worse, and with an increased amount of C contents in the marking material, the correct reading rate after heat-treatment is remarkably worse. Further, if the Fe contents in the marking material is increased, the correct reading rate after heat-treatment is raised. Although the reason for the above phenomena is not clear, it is assumed that since a mark produced with a marking material made of stainless material is black or blackish brown, the stainless material which has already been oxidized is formed on the glass plate. This oxide mark is considered as a compound oxide containing elements (Fe, Cr, Ni) of the marking material as primary components, or a mixed oxide, that is, a mixed oxide comprising an iron oxide, a chromium oxide and a nickel oxide. Further, these compound oxides or mixed oxides are oxides slightly

reduced and whose color is changed by oxygen when it is supplied in the heat-treatment. Since the correct reading rate is lowered as the amount of Ni is increased, it is presumed that the color change is mainly caused by the change of NiO_{1-x} to NiO which is included in the marked film. Further, with reference to a chromium oxide, the light-shielding-property (the characteristic of light shield) is affected a little by oxidization. Carbon in the marking material exerts a large influence on the mark quality. It is considered that carbon exists in the mark after marking is applied, but is gasified (carbon gas) by heat-treatment. This can be presumed by the fact that the mark formed from the marking material containing a large quantity of carbon such as SUS440A, S45C loses a light shielding property being subjected to the heat-treatment process.

According to the present embodiment, by using stainless steel or steel as the marking material, it becomes possible to produce QR Code, Data Code, Veri Code, or two-dimensional bar codes, or bar codes, each having quality which does not varied when subjected to heat-treatment. Particularly, when the stainless material not containing a large quantity carbon and nickel, like low carbon steel, martensite group or ferrite group stainless steel, is used as the marking material, mark quality does

not deteriorate through heat-treatment allowing code recognition of higher reliability. Further, since stainless steel and steel are widely used as structural material, allowing easy machining and grinding, being available at low cost, they contribute to lowering the market price of the marking material.

In the present embodiment, as shown in Fig. 1, the mark of the two-dimensional data code is formed through the first process for depositing the film to the glass plate and the second process for removing the deposited film, but in some cases, the mark is completed by only the first process without going through the second process. In the production line of a liquid crystal panel or a plasma display panel, in some cases, a somewhat large character is marked as the substrate manufacturing number to allow easy recognition. Also in this case, the marking quality of the marking material of the present invention does not deteriorate by heat-treatment, thereby serving to further the cost reduction of the marking material. Further in the present embodiment, a two-dimensional data code is used as the mark to review the recognition performance, however, QR Code, Veri Code, or a two-dimensional bar code which all belong to a minute code or a bar code can be used with sufficient heat-resistance, and hence it is apparent that the mark cost can be reduced

thereby.

It is to be noted that, in the present embodiment, SUS301, SUS304, SUS310S, SUS405, SUS430, SUS447J1, SUS403, SUS410 and SUS440A as well as S10C, S25C, S35C and S45C of the JIS (Japanese Industrial Standard) are used as the stainless material, however, other stainless material and steel material of the JIS can also be used. Further, the marking material of the present invention is not limited to the material of the JIS, but mass-produced steel or stainless steel of low price of a foreign standard can be used for the marking material of the present invention.

Further, the liquid crystal panel and the plasma display panel are not only heat-treated by the above temperature, but generally additionally subjected to chemical treatment such as etching in the manufacturing process. Therefore, the formed mark of the two-dimensional bar code or the bar code must be chemicals-proof. In the present embodiment, the marking materials of SUS301, SUS304, SUS310S, SUS405, SUS430, SUS447J1, SUS403, SUS410 and SUS440A as well as S10C, S25C, S35C and S45C are used as manufacturing marks of Two-dimensional Data Code, and the marks are heat-treated at 650 °C for 30 minutes, and then dipped for 10 minutes in 5 % hydrochloric acid, 10 % hydrochloric acid, and 5 % sodium hydroxide aqueous solution, respectively, to produce each

sample. After washing each sample with pure water, the correct reading rate of each sample is measured with reference to the Two-dimensional Data Code.

[Table 2]

marking material	Correct Reading Rate (%)				
	marking material	heat-treatment in atmosphere, 650°C- 30 min.	5% HCl	10% HCl	5% NaOH
SUS301	100	85	80	76	84
SUS304	100	82	77	73	81
SUS304L	100	76	70	68	75
SUS301S	100	60	60	60	60
SUS405	100	93	93	85	92
SUS430	100	89	89	87	88
SUS447J1	100	87	85	81	85
SUS403	100	94	94	90	92
SUS410	100	92	92	89	91
SUS440A	100	52	52	50	52
S10C	100	94	55	35	91
S25C	100	92	49	31	90
S35C	100	70	33	23	67
S45C	100	50	25	13	49

The correct reading rate of the Two-dimensional Data Code marks whose marking material are stainless steel show substantially the same results with reference to the case after heat-treatment, after being dipped in a hydrochloric acid, and after being dipped in a sodium hydroxide aqueous solution. As describe above, the Two-dimensional Data Code mark formed using stainless steel marking material is a strong mark being acid-proof and alkali-proof in addition to be heat-proof. Compared with a mark using steel marking material later described, a mark using

stainless steel as the marking material can be used in the manufacturing process of a common liquid crystal panel or a plasma display panel without any problems and capable of code recognition with high reliability. The mark according to the present invention described above is considered to have a mark film of compound oxides consisting of iron, chromium and nickel.

The Two-dimensional Data Code mark whose marking material is iron steel shows substantially no change in correct reading rate when dipped in sodium hydroxide aqueous solution, however, the correct reading rate becomes worse when dipped in hydrochloric acid. Particularly, when dipped in 10 % hydrochloric acid, the correct reading rate becomes worse remarkably, to less than 50 % of the value obtained after heat-treatment. However, the marking material of steel is effective in a process in which no acid solution is used.

According to the present embodiment, by using steel or stainless steel as a marking material, it becomes possible to provide a code mark with chemical resistance and high reliability.

(Embodiment 2)

As described above, a mark such as a Two-dimensional Data Code mark to be read by transmitted illumination is required to have uniform film thickness throughout the

mark. Fig. 2 is a view showing the change of the YAG laser power used in the first process of Fig. 1 through time, in the process of depositing a film to a glass plate. The YAG laser for marking use is generally applied with pulses of 1 to 10 KHz, and uniform marking becomes possible when the peak power of each pulse reaches the same value as shown in Fig. 2. However, according to operating conditions, the YAG laser may possibly occur power fluctuation as shown in Fig. 3. The reason for this power fluctuation is presumed to be mode fluctuation of the laser, fluctuation of the quantity of light of the Krypton arc lamp used as the excitation source of the laser, temperature variation of a coolant for cooling the YAG rod, instability of the Q switch driver for generating the pulse laser, defective adjustment of the optical system, or a defective control method for the first pulse killer at the marking starting point or the like. In recent years, although the performance of lasers have considerably improved, skilled technique is further required to obtain a laser oscillation of the stable power shown in Fig. 2, and it has been necessary to use the YAG laser with the knowledge that the YAG laser has power fluctuation of about $\pm 10\%$ at the minimum. In the case when marking shown in the present embodiment 1 (Fig. 1) is performed using a laser having power fluctuation as shown

in Fig. 3, since the amount of marking material on the glass plate for the mark in the first process varies with the fluctuation of the laser power, the film thickness of the mark also varies. When mark like this is read by a code reader using transmitted illumination, since the light-shielding-property is affected by unevenness of the film thickness, correct code reading is difficult. In the present embodiment, Two-dimensional Data Code marking is prepared by using a metal film formed on the glass plate, a laser transmittive body, and subjected to a reading recognition test to review the performance.

Fig. 4 shows a structure of embodiment 2 and a marking process thereof. In Fig. 4, the symbol 1a represents a metal film formed on glass plate 1, the numeral 2 a glass plate used as the material to be marked, 6a and 6b are chips to provide a gap between the marking material and the material to be marked (Fig. 4(a)). In the present embodiment, there is provided a gap of about $50 \mu m$. The marking method comprises the steps of scanning and applying a laser beam LA such as the YAG laser to metal film 1a through glass plate 2 to evaporate metal film 1a, thereby making a film M deposit to a specific area of glass plate 2, as shown in Fig. 4(b); successively separating glass plate 2 from glass plate 1 at a Z stage not shown and then scanning the laser beam

LA while controlling irradiation thereof by means of a Q switch in order to apply the laser beam LA to the predetermined part of the film M, thereby forming a predetermined figure as shown in Fig. 4(c). In the present embodiment, a Two-dimensional Data Code of 7 cells \times 7 cells is prepared.

The present embodiment uses chromium for the metal film, soda glass for glass plate 1, and fused silica for glass plate 2. The two-dimensional Data Code mark formed on glass plate 2 has a clear contrast and is easily recognizable after marking by using a data code reader.

Subsequently, in the process of Fig. 4(b), the quantity of light of a Krypton arc lamp, which is a laser excitation source, is intentionally varied during marking to form the fluctuated state of the laser power shown in Fig. 3, thus forming a Two-dimensional Data Code. The maximum power fluctuation rate at this time is decided as 50 % of the peak power. The Two-dimensional Data Code thus formed is easily recognizable by means of the data code reader in the same way as the Two-dimensional Data Code formed in the state with no power fluctuation resulting in the clear mark. When the metal film on the glass plate is used as the marking material in this way, since the heat capacity of the metal film is small compared to that of bulk material, all the metal film is

evaporated by the small laser power and then deposited on the glass plate. Therefore, the metal film is not affected by variation of the laser power, thereby being able to provide a code mark of high reading reliability. Further in the present embodiment, although a chromium films of a thicknesses of $0.1\ \mu\text{m}$, $0.2\ \mu\text{m}$, $0.3\ \mu\text{m}$, $0.5\ \mu\text{m}$, $0.7\ \mu\text{m}$, $1\ \mu\text{m}$, $2\ \mu\text{m}$, $3\ \mu\text{m}$, $5\ \mu\text{m}$ or $10\ \mu\text{m}$ is used as the metal film, all succeeded in forming a clear two-dimensional mark. However, in the case of film thickness of $3\ \mu\text{m}$ or more, a part of the chromium film on glass plate 1, which is not used for forming the code, sometimes strips off at the time of marking, and it is desirable that the film thickness thereof be $2\ \mu\text{m}$ or less for practical use. Further, it is apparent that a Two-dimensional Data Code being heat-and-chemicals-proof can be provided when stainless steel or steel is used as the metal film. Other metal film made of the marking material described in embodiment 1 may also be used.

In the present embodiment, although a marking material having a metal film deposited on the glass plate is used, inorganic material such as sapphire, quartz other than glass plate may be used as long as the laser beam transmits through the material. Although the YAG laser is also used in the present embodiment as the laser beam, if it matches the material to be marked, it is allowable to

use a carbon gas laser. For example, in the marking operation can be performed with the structure and the process shown in Fig. 4, if a silicon wafer is used as material-to-be-marked 2, organic matter such as paint or ink or metal such as Cr or gold or inorganic matter such as iron oxide, SiO_2 , silicon nitride is used as thin film 1a, and a silicon wafer is used as substrate 1 of thin film 1a, paint or ink, Cr or gold, iron oxide or SiO_2 or silicon nitride can be marked on silicon wafer 2. In the present embodiment, review of recognition performance is performed with reference to the mark expressed with the Two-dimensional Data Code, however, QR Code, Veri Code, or a two-dimensional bar code, each of which in a minute code, or bar code can be used as the code.

Embodiment 1 and embodiment 2 are executed for marking on a liquid crystal panel or a plasma display panel. However, the present invention is not limited to these uses, and if the material to be marked is glass, the present invention can be applied, for example, to window glass of a building, windshield glass of an automobile or a lens of optical equipment such as eyeglasses or a microscopes.

A marking material can be processed by forming a thin film on the surface of a transparent body or a laser transmittive body in each of the other embodiments.

(Embodiment 3)

Fig. 5 shows, in sectional views and plan views, a marking method with reference to embodiment 3 of the present invention. In the process, glass plate 2 is placed on metal plate 1 made of chromium (a). Then, by using a YAG laser apparatus, the focus of the laser beam LA is placed on surface 1a of metal plate 1 matched glass plate 2, and the 40 μ m diameter laser beam of first laser power (in the present embodiment, 45 W or 45 mJ/pulse or more) scans the area (shown in the Fig. 5(b) by a dotted line, 2 mm square) with an interval of 50 μ m (b). When glass plate 2 is separated from metal plate 1, a chromium thin film MC deposited to glass plate 2 is found in the area scanned by the laser beam (c). Further, 40 μ m diameter laser beam LA is applied by second laser power on the chromium thin film MC deposited on glass plate 2 and scanned to form a desired character, diagram or symbol (d). The chromium thin film MC irradiated by the laser beam effected by the second laser power (in the present embodiment, 11 W or 11 mJ/pulse or more) is removed to form a character, a diagram or symbol 3. The formed character, diagram or symbol 3 may become the portion where the laser beam is applied and transmits therethrough or a portion on which the chromium thin film MC remains.

Further, in the second process of the present

embodiment (Fig. 5(d)), glass plate 2 is marked after being separated from metal plate 1. However, by selecting a scanning method and a laser power appropriately, it is possible to mark to glass plate 2 holding metal plate 1 deposited thereto. In this marking method, through pulsed irradiation and concurrent scanning of the laser beam applied with an appropriate speed keeping the laser power in a range of 5 - 10 mJ/pulse, the chromium thin film deposited in the first process is removed being instantaneously heated by the pulsed laser beam. By processing in this way, the same mark as that of the above embodiment can be formed.

In the present embodiment 3, character, diagram or symbol 3 having the resolution of a laser beam diameter of 40 μ m is formed as in embodiment 1. However, since the glass and the metal is closely contacted, there is a possibility that glass plate 2 may crack or break, and hence it is desirable to have a gap between metal plate 1 and glass plate 2 as shown in embodiment 1.

(Embodiment 4)

In the present embodiment, a Two-dimensional Code as shown in Fig. 6 is made by using titanium as the material according to the same procedure as that of embodiment 1. Results of marking are shown as parameters of the first laser power and the second laser power. In Fig. 6, a code

is composed of 20 cells arranged widthwise and 20 cells arranged lengthwise, in total 400 cells. An L pattern is composed of the left side and the bottom side, and the right side and the upper side pattern have white and black marks arranged therein alternately. Both configuration a pattern with which a two-dimensional code reader discriminates the position of the code. 18 horizontal and 18 lengthwise directional cells amounting to 324 cells are surrounded by the above four sides and record data such as a diagram, a numeral, or an English character. In other words, the Two-dimensional Code of the present embodiment can record data of 324 bits.

A procedure for manufacturing the ATwo-dimensional Code comprises the steps of scanning the laser beam in the first process by a $50\ \mu\text{m}$ interval as shown by the arrow mark in Fig. 7. Then, forming each square cell of a white ground by scanning and applying the laser beam to form a pattern of a \square type as shown in Fig. 8 by the second laser power (10 W in the present embodiment) in the second process, thereby entering data in 400 cells in total. By scanning the laser beam while keeping the first laser power constant (in the present embodiment, 30 W), the irradiation energy power is raised to increase the amount of metal evaporated at the place where the laser beam turns, corresponding to the left and right sides shown in

Fig. 7 (an enlarged part of Fig. 7), thereby increasing the amount of metal deposited to the glass plate, resulting in a Two-dimensional Code having a thick film at the turning point. Therefore, two problems are raised. First, in the second process, the film on the glass plate can not be removed sufficiently at the thick portion, consequently producing speck. Second, since the L pattern formed in the second process requires linking □ patterns together and drawing takes a long time.

Therefore, as another embodiment, in the first process in Fig. 7, the laser beam scans to form segment 8a, 8b with a first laser power of 30 W and segment 9 with a first laser power 25 W. Also in the second process, for a part corresponding to the L character pattern, as shown in Fig. 9, it was formed by one stroke with a second laser power of 20 W. As a result, in the first process, an almost uniform film is formed with the thickness within $\pm 3\%$ in variation. Also in the second process, the drawing time for the L pattern is reduced to 1/5. It is to be noted that in the present embodiment, Two-dimensional Codes are made in the first process and the second process by means of two different laser powers, respectively, however, the laser power of three or more grades may be used according to the patterns of characters, diagrams or symbols to be made.

As described above, in the first and second processes, two or more laser power utilization makes it possible to obtain speckless mark. The marking time is also reduced.

(Embodiment 5)

In the present embodiment, according to the same procedure as that of embodiment 1, titanium was used as the material, and the Two-dimensional Code shown in Fig. 6 was manufactured using the first and the second laser powers as parameters. As parameters a first laser power of 20, 30, 40, 50, 60, 70 w, and a second laser power of 10, 15, 20, 25, 30 w are used. Fig. 10 shows a relation between film thickness of a titanium compound formed on the glass plate and the first laser power. After forming a film with each of the above first laser powers, a two-dimensional code test is performed by applying the laser beam with each of the above second laser power. Table 3 gives the test results.

[Table 3]

second laser power \ first laser power	20W	30W	40W	50W	60W	70W
10W	×	○	○	○	○	×
15W	×	○	○	○	○	×
20W	×	○	○	○	○	○
25W	×	○	○	○	○	○
30W	×	×	○	○	○	○

Manufacturing results were judged by the sample reading test performed with a two-dimensional code reader. In table 3, O represents that the sample is readable, and X represents that the sample is unreadable.

With a first laser power of 20W, since there is scarcely any film deposited to the glass plate, no readable Two-dimensional Code is made. Further, since the Two-dimensional Code made by a first laser power of 30W and a second laser power of 30W has a lot of speck, it is unreadable by the two-dimensional code reader. Further, with a first laser power of 70W and a second laser power of 15W or less, the film cannot be perfectly removed, so the mark cannot be read by the two-dimensional code reader. From the above results, it is obvious that a readable Two-dimensional Code can be made when the first laser power is higher than the second laser power.

(Embodiment 6)

Another marking method will be described in this embodiment. Fig. 11 shows the marking process of this embodiment. In this process, chips 6a and 6b of 100 μ m in thickness are placed on metal plate 1 made of titanium, and glass plate 2 is placed on the chips, making a gap of 100 μ m between metal plate 1 and glass plate 2 (a). By using a YAG laser device and adjusting the focus of the

laser beam LA on metal plate 1, a laser beam is scanned to a desired area (2 mm square) with a 50 μ m interval by means of the first laser power (in the present embodiment, not less than 30 W or 30 mJ/pulse). The metal of metal plate 1 irradiated by the laser beam LA is heated and melted, partially evaporated, ionized or turned to plasma. Metal evaporated, ionized or turned to plasma chemically reacts with the gas component in the gap between metal plate 1 and glass plate 2 to produce titanium monoxide, dititanium trioxide which deposit to surface 2a of glass plate 2. Through the first process described above, a black film M of 0.2 μ m in thickness is formed on glass plate 2 in an area of 2 mm square (b). Subsequently in the second process (c), glass plate 2 is separated from metal plate 1. In applying the laser beam to film M on surface 2a of glass plate 2 formed in the first process by a second laser power (in the present embodiment, less than 10 W or 10 mJ/pulse), the laser beam is scanned to form a predetermined character, diagram or symbol. At this time, the laser beam is applied from the direction reverse to that in (b). Upon receiving the irradiation of the laser beam, the laser irradiated portion of the film M comprising titanium monoxide, dititanium trioxide is removed from surface 2a of glass plate 2. Thus, characters, diagrams or symbols 3 of a clear contrast are

marked on glass plate 2.

The beam diameter of the laser beam used in the present embodiment is $40\text{ }\mu\text{m}$, and it has succeeded in drawing a diagram having the resolution of the beam diameter. It is noted that when marking is performed according to the prior art using a laser beam of $40\text{ }\mu\text{m}$ beam diameter, the resolution of the diagram is $50\text{ }\mu\text{m}$ or more. In other words, according to the present embodiment, it is possible to form a minute mark having the same resolution as that of the laser beam diameter. Further, in the present embodiment, two independent laser sources are used in a first process and a second process, however, if a process for turning glass plate 2 by 180° is provided between the first process and the second process, the same marking as that of the present embodiment can be made with one laser source.

(Embodiment 7)

Another marking method will be described in this embodiment. Fig. 12 shows the marking process of the embodiment. In the process, chips 6a and 6b of $100\text{ }\mu\text{m}$ in thickness are placed on metal plate 1 made of titanium, and glass plate 2 is placed on the chips to form a gap of $100\text{ }\mu\text{m}$ between metal plate 1 and glass plate 2 (a). By using a YAG laser device and adjusting the focus of the laser beam LA on metal plate 1, a laser beam is scanned to

a desired area (2 mm square) with a 50 μ m interval by the first laser power (in the present embodiment, not less than 30 W or 30 mJ/pulse). Therefore, the metal on metal plate 1 irradiated by the laser beam LA is heated and melted, partially evaporated, ionized or turned to plasma. Metal evaporated, ionized or turned to plasma chemically reacts with the gas component in the gap between metal plate 1 and glass plate 2 to produce titanium monoxide, dititanium trioxide which then deposit to surface 2a of glass plate 2. Through the first process described above, a black film M of 0.2 μ m thickness is formed on glass plate 2 in an area of 2 mm square (b). Subsequently in the second process (c), by applying the laser beam to the film M on surface 2a of glass plate 2 formed in the first process by the second laser power (in the present embodiment, less than 0.1 W or 0.1 mJ/pulse), the laser beam is scanned to form a predetermined character, diagram or symbol. At this time, the laser beam is scanned in stepping motion so that the laser beam can stay at a predetermined spot for one to two seconds. Upon receiving the irradiation of the laser beam, the laser irradiated portion of the film M comprising titanium monoxide, dititanium trioxide are locally heated by absorption of the laser energy, thereby changing to titanium dioxide MO. Since the portion changed to titanium dioxide becomes

transparent, it becomes possible to perform marking in the same way as in embodiment 1. Here, the change into titanium dioxide to become transparent is called transformation. Through the above process, characters, diagrams or symbols 3 of a clear contrast are marked (d).

The diameter of the laser beam used in the present embodiment is 40 μ m, and it succeeded in drawing a figure having the resolution of the beam diameter. It is noted that when marking is performed according to the prior art using a laser beam of 40 μ m beam diameter, the resolution of the diagram is 50 μ m or more. In other words, according to the present embodiment, it is possible to form a minute mark having the same resolution as the laser beam diameter. Further, in the present embodiment, although titanium is used as the marking material, alloy or a metal compound or a compound containing titanium may be used. Further, it is obvious that the same result as in the present embodiment can be obtained using the material by which film M is formed by heat in the first process and the color of the film M is thermally changed later. It was found in another test that the color of black silver oxide can be changed to lustrous white by depositing the black silver oxide to the glass in the first process and applying the laser beam in the second process to the silver oxide deposited in the first process

for reducing and changing to metal silver.

(Embodiment 8)

Fig. 13 shows the process of the marking method of the present invention in perspective views and sectional views. In the process, chips 6a and 6b of 100 μ m in thickness are placed on metal plate 1 made of titanium, and then glass plate 2 is placed on the chips thereby forming a gap of 100 μ m between metal plate 1 and glass plate 2 (a). Then, by using a YAG laser device and adjusting the focus of a laser beam LA on metal plate 1, and scanning the surface of metal plate 1 under a predetermined laser irradiation condition at a fixed speed to produce desired characters, diagrams and symbols (b). The metal of metal plate 1 irradiated by the laser beam LA is heated and melted, partially evaporated, ionized or turned to plasma. Metal evaporated, ionized or turned to plasma chemically react with the gas component in the gap between metal plate 1 and glass plate 2, producing a substantially black film M on glass plate 1 (c). As a result of an analysis of the film M by X-ray photoelectron spectroscopy, a large quantity of Ti^{2+} , Ti^{3+} and O^{2-} were confirmed and also some N^{3-} was detected. Therefore, it is presumed that the greater part of the mark formed on the glass plate is composed of titanium monoxide, dititanium trioxide and that a small quantity of titanium nitride

also exists therein .

Fig. 14 shows data of the surface configuration of the glass after marking is applied, the data being measured by means of a surface roughness instrument. It was confirmed that there was deposited matter of about 0.3 μ m in the marking portion of the glass surface. According to the marking method of the present invention, since glass plate 2 is separated from metal plate 1, the metal does not directly contact the surface of glass plate 2 even if the metal is melted being irradiated by the laser beam, and hence glass plate 2 is protected against generation of a crack and even when worst comes to worst, perfectly protected against breaking. Further, no dent is formed in glass plate 2, the bending strength of the glass plate is not in the least deteriorated. Besides, since the mark is black, it is easy to recognize and a code is recognizable by a reader such as a bar code reader. It is noted that although a glass plate is used in the present embodiment, it is clear that the same effect will be obtained by using a transparent or a translucent body for a laser beam.

Further, since a clear square mark can be formed on the glass plate according to the present embodiment, a film M can be formed by applying the present embodiment to the first process of embodiment 1, 3, 6 and 7.

(Embodiment 9)

Although the previous embodiments used titanium as the metal plate, the present embodiment uses titanium-niobium alloy, titanium-zirconium alloy, titanium monoxide, titanium hydride, titanium/titanium monoxide compound to apply marking in the same way to the glass plate. Table 4 gives the color of markings obtained by using each of the above material, valence of the titanium atoms of the marked portion obtained by X-ray photoelectron spectroscopy, and a titanium compound identified from the result of X-ray diffraction.

[Table 4]

material	marking color	valence of titanium	Ti compound to be indentified
titanium-niobium alloy	blackish gray	Ti^{4+} , Ti^{3+}	TiO Ti_2O_3
titanium-zirconium alloy	dark	Ti^{3+}	Ti_2O_3 TiO
titanium monoxide	blackish purple	Ti^{3+}	Ti_2O_3
titanium hydride	black	Ti^{4+}	TiO
titanium-titanium monoxide	black	Ti^{3+}	TiN Ti_2O_3

As a result, when marking is performed by using titanium-niobium alloy, titanium-zirconium alloy, titanium monoxide and dititanium-trioxide are detected. When titanium monoxide is used for marking, dititanium-

trioxide is detected. When titanium hydride is used, titanium monoxide is detected. When titanium-titanium monoxide compound is used, a large quantity of dititanium-trioxide and a small quantity of titanium monoxide are detected. The color when titanium-niobium alloy is used for marking is blackish gray, titanium-zirconium alloy is dark, titanium monoxide is blackish purple, titanium hydride is black, and titanium/ titanium monoxide compound is also black. All markings prepared by using the above materials have sufficient contrast for visual recognition. When a bar code was prepared under the above marking conditions and a code recognition test was performed with a bar code reader, every test piece proved to be readable, except titanium-zirconium alloy marking.

Therefore, according to the present embodiment, it is clear that it is possible to provide a sufficiently recognizable mark difficult to attain in the prior art by using a material containing titanium. Further, according to the result of measuring the surface configuration, since there is no damage such as a dent in the glass surface, it is possible to provide a mark with high mechanical strength.

(Embodiment 10)

In the present embodiment, marking is performed under a predetermined oxygen partial pressure using the

apparatus shown in Fig. 15. In Fig. 15, the numeral 7 denotes a chamber wall made of stainless steel, 8 a chamber base made of stainless steel, 9 a gas introduction port, 10 a window made of quartz glass which transmits a laser beam into the chamber, 11 a zirconia gas sensor for measuring concentration of oxygen in the chamber, 12 a gas exhaust port, 13 an O-ring, 14 a pressure gauge for measuring the pressure in the chamber, 15 an oxygen gas cylinder, 16 a cylinder for argon gas which serves as carrier gas, 17 a valve, 18 a gas flow rate controller, 19 a marking chamber. Glass plate 2 is placed on titanium plate 1 forming a gap therebetween by interposing chips 6a, 6b of 100 μ m in thickness. The laser beam is applied while scanning so as to form a figure of a bar code and a character under the predetermined gas pressure. First, Oxygen gas is selected as reaction gas and argon is selected as carrier gas, and then setting oxygen partial pressure to 1, 2, 5, 10, 20, 40, 80, 160, 380, 760, 1140, 1520 torr, marking is performed. After finishing marking, the mark has metallic luster with the oxygen partial pressure 1 torr, and then the atom detected by the X-ray photoelectron spectroscopy is only titanium having hardly any oxygen. That is, it is considered that, when the oxygen partial pressure is 1 torr, titanium evaporated by the laser beam LA deposits to glass plate 2 in the state

of metal. When the oxygen partial pressure is 2 torr, the mark shows black color with slight metallic luster, and Ti^{2+} and O^{2-} are detected from the result of X-ray photoelectron spectroscopy. In other words, it is presumed that, with an oxygen partial pressure of 2 torr, the part of titanium evaporated by the laser beam has reacted into TiO and mixed with metal titanium. With the oxygen partial pressure of 5 to 760 torr, the mark is black and Ti^{3+} , Ti^{2+} and O^{2-} are detected from the result of the X-ray photoelectron spectroscopy. In other words, it is presumed that, with an oxygen partial pressure of 5 to 760 torr, the titanium evaporated by the laser beam has reacted into TiO , Ti_2O_3 and mixed with metal titanium. With an oxygen partial pressure of 1140 torr or more, the mark is white and Ti^{4+} and O^{2-} are detected from the result of the X-ray photoelectron spectroscopy. Specifically, it is presumed that, with an oxygen partial pressure of 1140 torr or more, the titanium evaporated by the laser beam has oxidized into TiO_2 and exists as TiO_2 being mixed with metal titanium.

As described above, according to the present embodiment, it is possible to provide a mark with a visually recognizable contrast under an oxygen partial pressure of 5 to 760 torr. Further, with reference to bar code figures, it was found that a clear mark is provided

which can be read by a bar code reader without fail.

Further, in the present embodiment, marking is performed in the same way using, in addition to alloy and compounds containing titanium atom, metal such as silver, gold, cobalt, chromium, copper, iron, manganese, molybdenum, niobium, nickel, lead, palladium, platinum, ruthenium, silicon, tin, vanadium, tungsten, zinc, and zirconium, alloy such as Ni-Co or Cu-Ni, an intermetallic compound of Nb_3Sn , a metal compound of Fe_3O_4 , and a compound of $CrO-Co_2O_3$ and Fe_2O_3-MgO . As a result, a substantially black mark on the glass surface is successfully formed when marking is performed using silver, cobalt, chromium, copper, manganese, niobium, nickel, palladium, platinum, ruthenium, vanadium, Ni-Co, Cu-Ni, Nb_3Sn , Fe_3O_4 , $CrO-Co_2O_3$ and Fe_2O_3-MgO . Moreover, when gold is used, a mark of lustrous red, when iron or tungsten is used, a mark of dark brown, with molybdenum a mark of gray, with lead a mark of reddish yellow, and with tin a mark of blue are formed on the glass plate. However, when silicon, zinc or zirconium is used, the color of the mark is white. In other words, according to the present embodiment, if the matter to be evaporated upon receiving laser irradiation can form a colored reaction product through reaction with oxygen, then a recognizable mark can be produced. Further, since the result of measuring the surface configuration

shows no damage such as a dent in the glass surface, a mark with high mechanical strength can be provided.

Further, it is noted that a film M can be effectively formed by applying the marking material used in the present embodiment. That is, metal such as gold, silver, cobalt, chromium, copper, manganese, niobium, nickel, lead, palladium, platinum, ruthenium, silicon, tin, vanadium, tungsten, and alloy made of Ni-Co or Cu-Ni, an intermetallic compound of Nb_3Sn , a metal compound of Fe_3O_4 , and a compound of $CrO-Co_2O_3$ and Fe_2O_3-MgO , can be applied to the first process of embodiment 1, 3, 6 and 7.

(Embodiment 11)

Oxygen is used as the reaction gas in embodiment 10, however, nitrogen is used as the reaction gas in the present embodiment for the study of marking. The marking apparatus employed has substantially the same structure as that used in embodiment 10. In Fig. 15, a cylinder for nitrogen gas, which serves as the reaction gas, is used in place of oxygen gas cylinder 15, and gas chromatography for measuring the concentration of nitrogen gas is used as sensor 11. Glass plate 1 is placed on metal plate 2 made of titanium, chromium, niobium, or tantalum interposing chips 6a, 6b of $100\ \mu m$ therebetween for forming a gap. A laser beam is applied while scanning so as to form a figure of a bar code and a character under a nitrogen gas

partial pressure of 1, 2, 5, 10, 20, 40, 80, 160, 380, 760, 1140, 1520 torr. As the result, with a nitrogen gas partial pressure 380 torr or more, titanium shows a mark of lustrous bronze, chromium shows a brown mark, niobium and tantalum show a black mark. That is, a mark with a contrast for allowing visual recognition can be provided not only with oxygen reaction gas but with nitrogen reaction gas.

Further, as for a bar code figure, it was found that a clear mark can be provided and read by a bar code reader without fail. Further, since the result of measuring the surface configuration shows no damage such as a dent in the glass surface, a mark with high mechanical strength can be provided.

It is to be noted that a film M can also be formed by applying the marking material proved to be effective in the present embodiment, that is, titanium, chromium, niobium and tantalum, to the first process of embodiment 1, 3, 6 and 7.

(Embodiment 12)

In the present embodiment, using the apparatus shown in Fig. 15, marking was tested under an oxygen partial pressure of 152 torr while maintaining the gap between glass plate 1 and metal plate 2 at 1, 2, 3, 5, 10, 20, 30, 50, 100, 150, 190, 200 and 300 μ m. According to the

result, the mark has metallic luster for a gap of $1\ \mu\text{m}$, a black mark with metallic luster for a $2\ \mu\text{m}$ gap, a black mark for 3 to $190\ \mu\text{m}$ gap, and a milky white mark for a $200\ \mu\text{m}$ gap. Specifically, with a narrow gap, metal is not oxidized and deposits to the surface of glass plate 1 without being changed. With 2 to $190\ \mu\text{m}$ gap, evaporated titanium forms TiO , Ti_2O_3 through oxidizing reaction and deposits to the surface of glass plate 1, and with a broad gap, a dent is formed in the glass surface as in the prior art and the marked portion looks milky white.

As described above, according to the present embodiment, a clear mark is formed with a gap between $2\ \mu\text{m}$ and less than $200\ \mu\text{m}$. Further, various marking tests have been tried using several metals other than titanium while varying the gap, and clear marks were successfully obtained using very similar gaps. Moreover, since the result of measuring the surface configuration showed no damage such as a dent on the glass surface, a mark with high mechanical strength can be provided. Further, as described above, although titanium in the metal state deposits to the glass surface when the gap is $1\ \mu\text{m}$, the metal plate and the glass plate do not directly contact each other because of the gap and marking can be finished without giving a thermal shock to the glass plate. Due to this structure, after marking is completed, no crack is

generated in the glass plate and no glass plate is broken, thereby eliminating the problem of the prior art (Japanese Patent Laid-open No. 008634/1994).

(Embodiment 13)

In the present embodiment, it was tried to form marking by applying the laser beam to an indium-tin compound oxide film (hereinafter referred to ITO film) coated on a glass plate with a predetermined laser power (in the present embodiment, 0.08W or less) and by scanning the laser beam so as to form a specific character. In a portion of the ITO film irradiated by the laser beam with the power of 0.02 to 0.08 W, scarcely any change can be detected with the naked eye. However, when the portion of the ITO film irradiated by the laser beam through a band pass filter for transmitting light with wavelength of 700 to 800 nm was checked, it was found that the reflection light is remarkably reduced, and the portions of the ITO film irradiated and not irradiated by the laser beam make a clear contrast which can be easily recognized. That is, the portion irradiated by the laser beam looks black because the light transmits therethrough, and the portion not irradiated by the laser beam looks white because of reflecting the light, thereby producing a mark with a clear contrast. In the present embodiment, characters are recognized due to reflection light, but they can also be

recognized in the same way with transmission light. In recognition due to the transmission light, the portion irradiated by the laser beam looks white because the light transmits therethrough, and the portion not irradiated by the laser beam looks black because the light is reflected, thereby producing a mark with a clear contrast. Therefore, if the reflexivity or the transmittivity of the film on the glass plate is varied by irradiation of the laser beam, a recognizable mark can be obtained. In the present embodiment, the laser beam is applied to the ITO film on the glass plate for varying the reflexivity and the transmittivity, however, if a film made of metal, alloy, a metallic compound or a compound of these matters other than the ITO film is used and the reflexivity as well as the transmissivity of the film can be varied by irradiation of the laser beam, it is evident that the same effect will be obtained. Further, by applying the present embodiment to the second process of embodiment 1, a mark of a clear contrast was successfully obtained.

CLAIMS

1. A method for marking materials using a material to be marked and a marking material consisting of a transparent body or a laser transmittive body, comprising;

a first process of placing the surface of said material to be marked and the surface of said marking material together with a desired gap therebetween, vaporizing said marking material by irradiating it through said material to be marked with a beam of a first laser power while scanning with the laser beam, and depositing a deposit vaporized from said marking material to a predetermined portion of said material to be marked; and

a second process of removing or denaturalizing said deposit deposited to the surface of said material to be marked with a laser beam of a second laser power; whereby patterns of characters, diagrams or symbols are formed on said material to be marked.

2. The method for marking materials according to claim 1, wherein the laser power of said first laser power is greater per unit area than the laser power of said second laser power.

3. The method for marking materials according to claim

1, wherein said deposit is subject to direct irradiation in said second process without passing said laser beam through said material to be marked.

4. The method for marking materials according to claim 1, wherein the deposit caused to degenerate by irradiation by said second laser power is changed in color or to made transparent by heating.

5. A method for marking materials in which the material to be marked and the marking material consist of a transparent body or a laser transmittive body, comprising the process of:

forming patterns of characters, diagrams or symbols on said material to be marked by placing the surface of said material to be marked and the surface of said marking material together with a desired gap therebetween;

vaporizing said marking material by irradiating it through said material to be marked with a laser beam while scanning with the beam; and

depositing a deposit vaporized from said marking material to a predetermined portion of the material to be marked; whereby

said patterns of characters, diagrams or symbols are formed by reacting the gas existing in said gap with the

vaporized marking material due to the evaporation of said marking material by said laser beam and the reaction product deposited to the desired portion of the material to be marked.

6. The method for marking materials according to claim 5, wherein the gas existing in said gap is one of either oxygen or nitrogen or both.

7. The method for marking materials according to either claim 1 or claim 5, wherein said gap is 2 μ m or more and less than 200 μ m.

8. A method for marking materials using a material to be marked and a marking material consisting of a transparent body or a laser transmittive body comprising the process of forming patterns of characters, diagrams or symbols by placing together the surface of said material to be marked with the surface of said marking material with a desired gap therebetween, vaporizing said marking material by irradiating it through said material to be marked while scanning with a laser beam and depositing a deposit formed from said marking material to a predetermined portion of said material to be marked, whereby

said patterns of characters, diagrams or symbols are formed by changing the transmittivity or reflectivity of the deposit formed from the marking material by laser beam irradiation.

9. A marking material for use in any one of claims 1, 5 and 8, wherein the marking material used is a metal, an alloy, an intermetallic or metal compound, or a compound containing at least one of said metal, alloy and intermetallic or metal compound.

10. The marking material of claim 9 wherein the thin film formed on the surface of the transparent body or laser transmittive body is of a thickness of 10 μ m or less and preferably from 0.1 μ m to 2 μ m.

11. A marking material for use in either claim 1 or 5 wherein the marking material is steel or stainless steel.

12. A marking material for use in claim 1 or 5 wherein the marking material is either a martensite or a ferrite stainless steel, or low carbon steel, and preferably steel with a carbon content of 0.25 % or less.

13. The marking material according to claim 11 or 12,

wherein the marking material is a thin film formed on the surface of a transparent body or laser transmittive body.

14. The marking material according to claim 13, wherein thickness of said thin film is 10 μ m or less, and preferably from 0.1 μ m to 2.0 μ m.

15. The marking material according to any one of claims 9 to 14, wherein the pattern formed is QR Code, Data Code, Veri Code, a two-dimensional code, or a bar code.

ABSTRACT

A method of marking materials, wherein the surface of a marking material and the surface of a material to be marked are matched, a laser beam is applied to the marking material while scanning the beam to form a mark, the material to be marked consisting a transparent body on which a pattern of a character or the like is formed. The method for marking comprises a first step of applying the laser beam to the marking material with the first laser power to evaporate the marking material and make them deposit to a predetermined part of the material to be marked, a second step of applying a laser beam to the deposited matter on the material to be marked with a second laser power to remove or transform the deposited mater. Steel or stainless steel is used as the marking materials.

Fig. 1 (a)

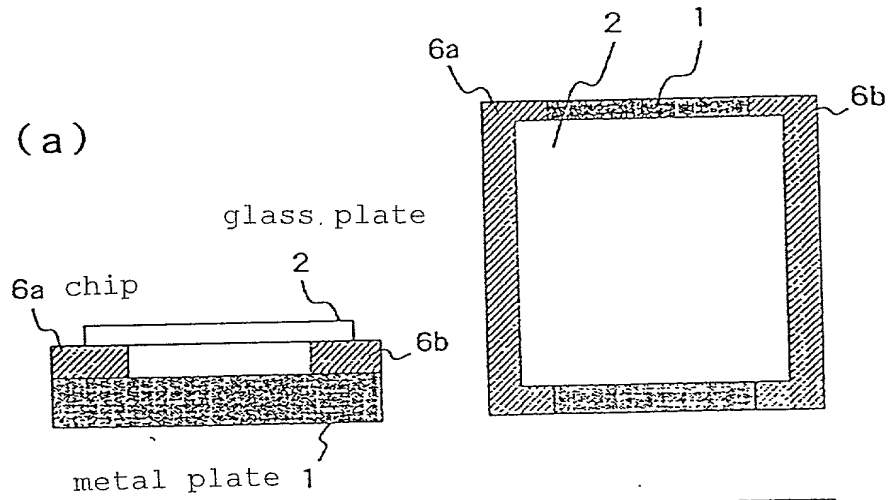


Fig. 1 (b)

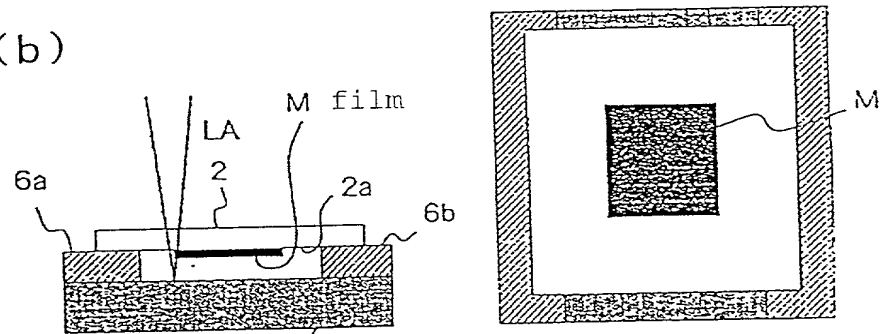
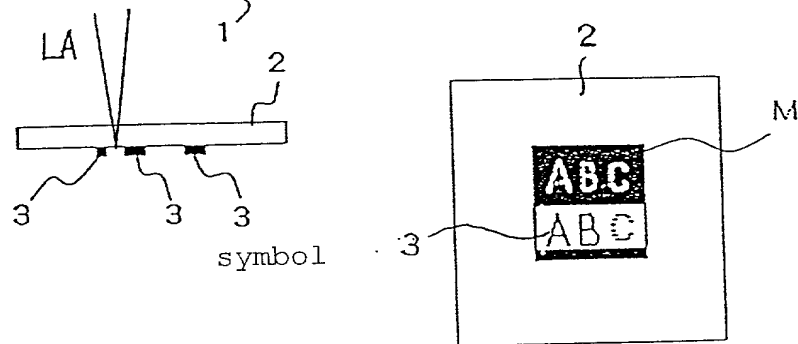


Fig. 1 (c)



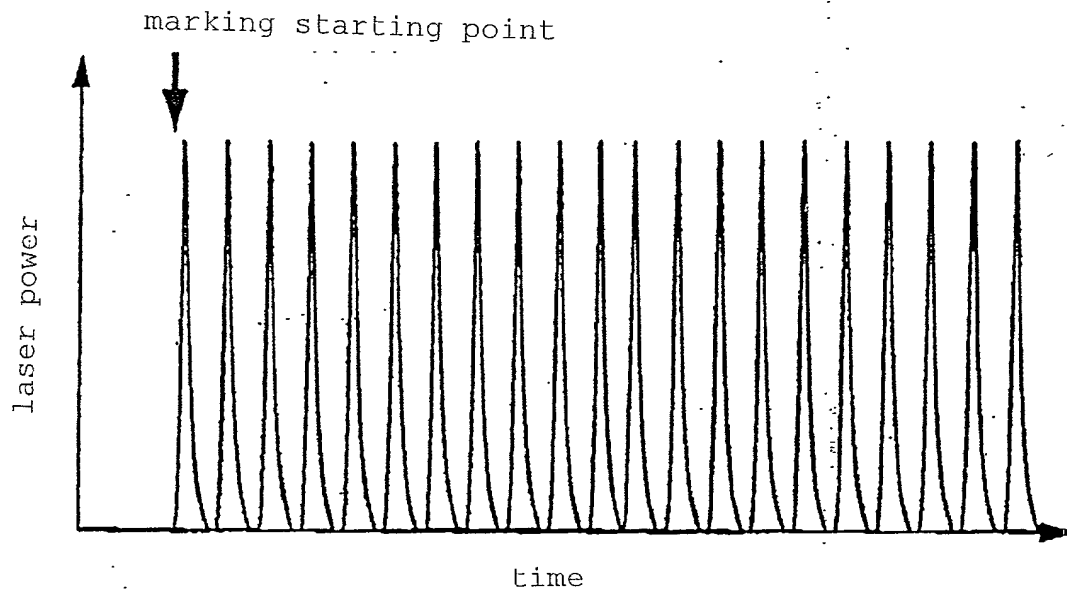


Fig. 2

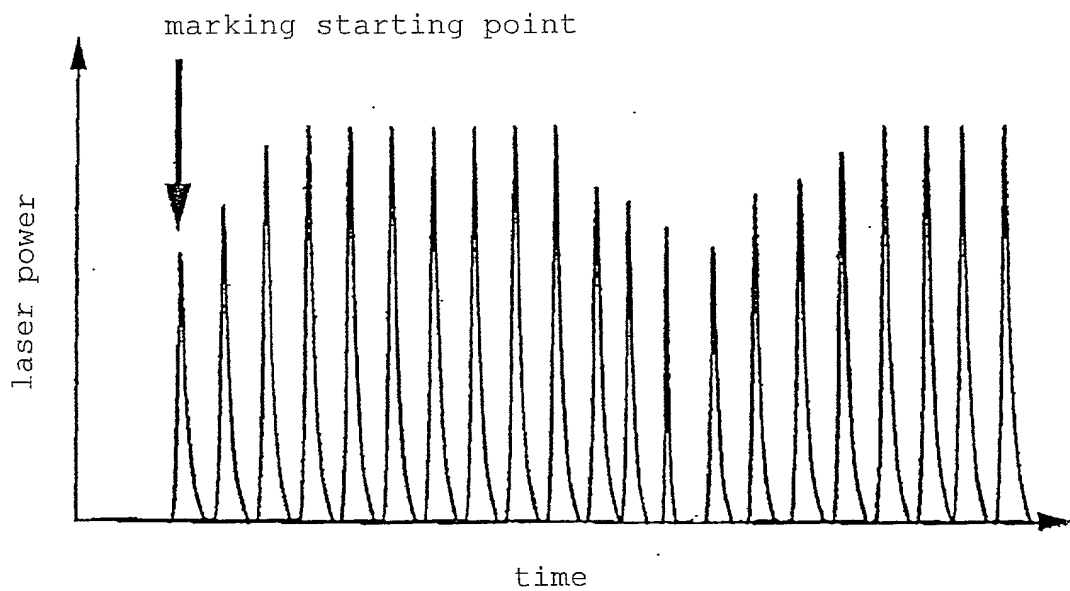


Fig. 3

Fig. 4 (a)

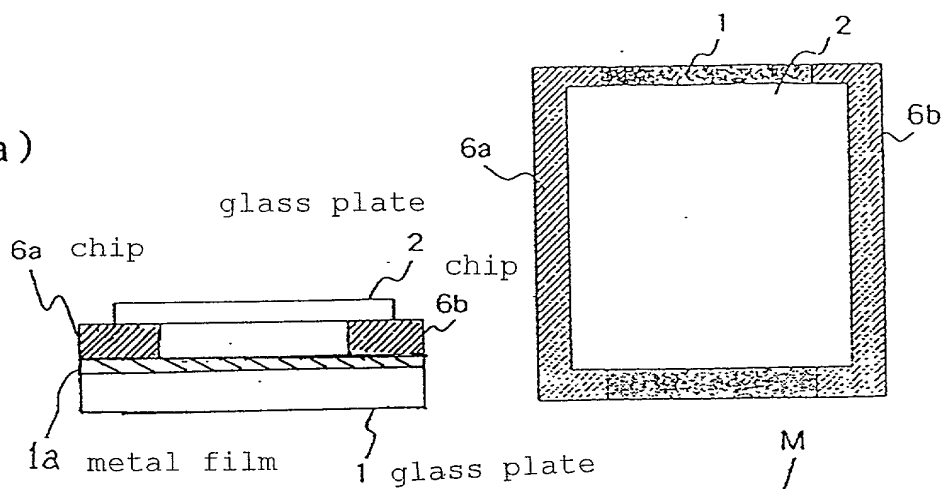


Fig. 4 (b)

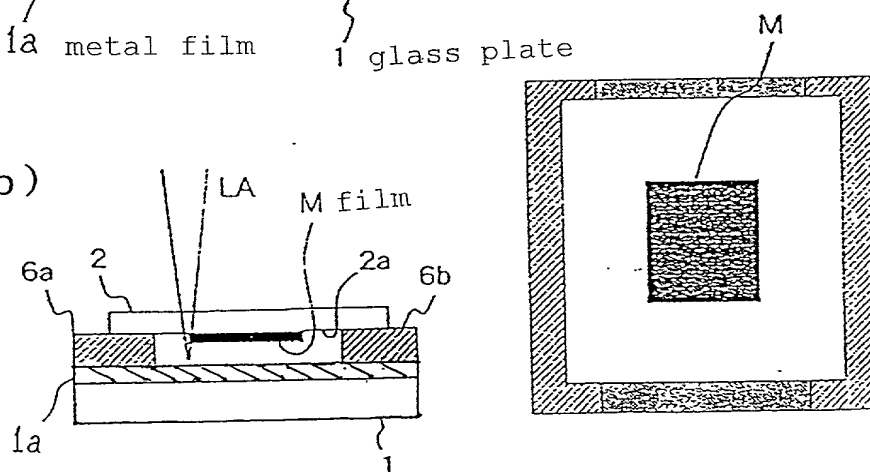


Fig. 4 (c)

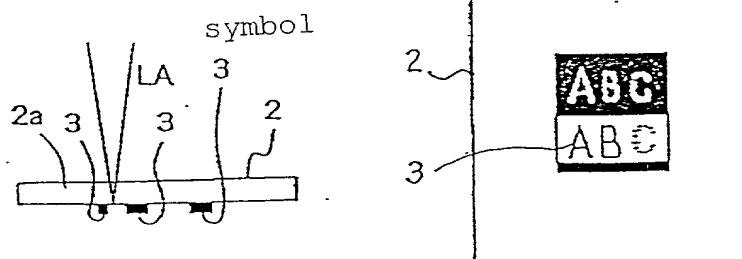


Fig. 5 (a)

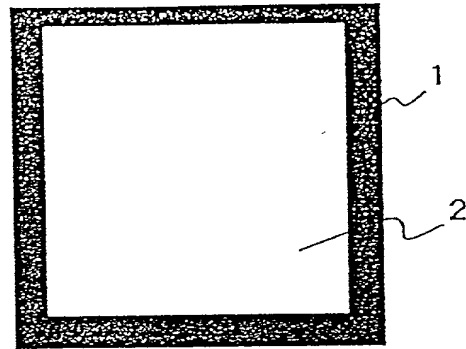
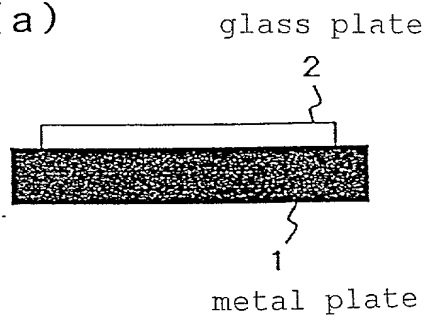


Fig. 5 (b)

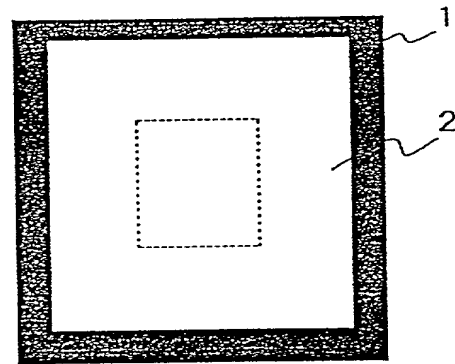
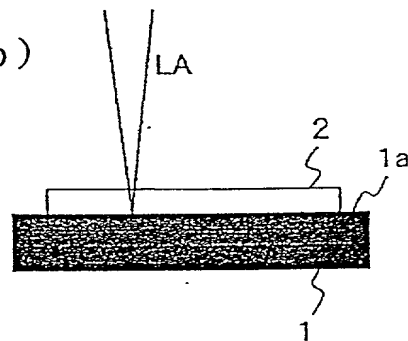


Fig. 5 (c)

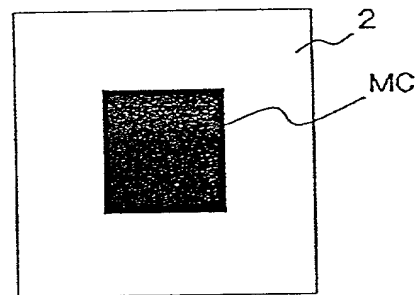
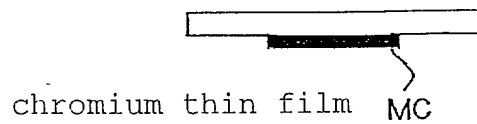


Fig. 5 (d)

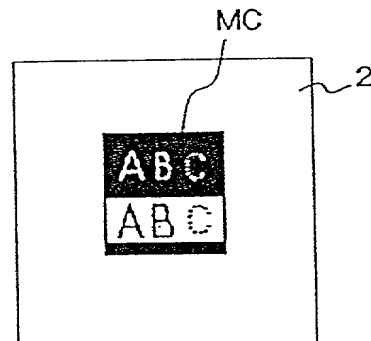
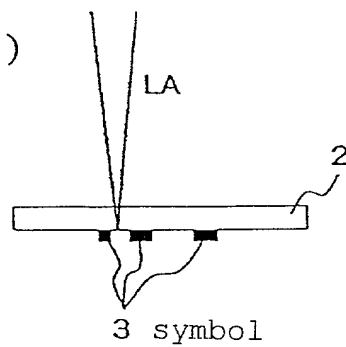


Fig. 6

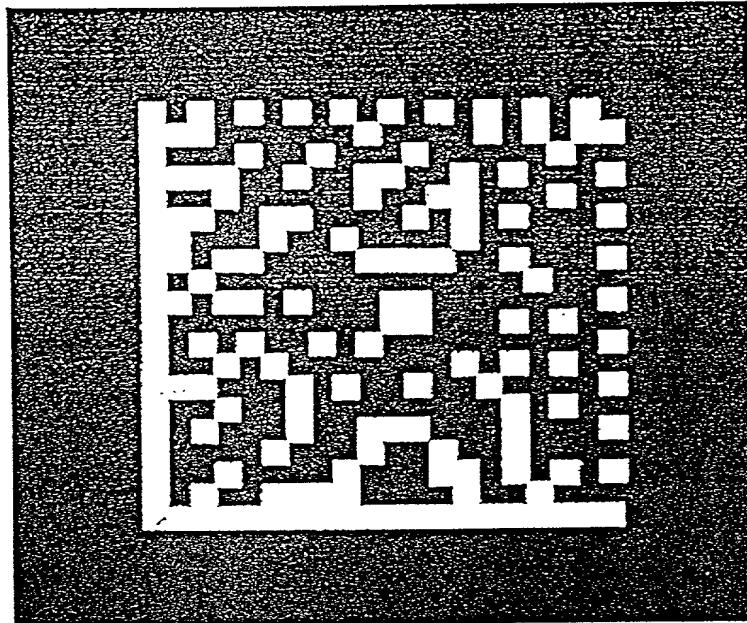


Fig. 7

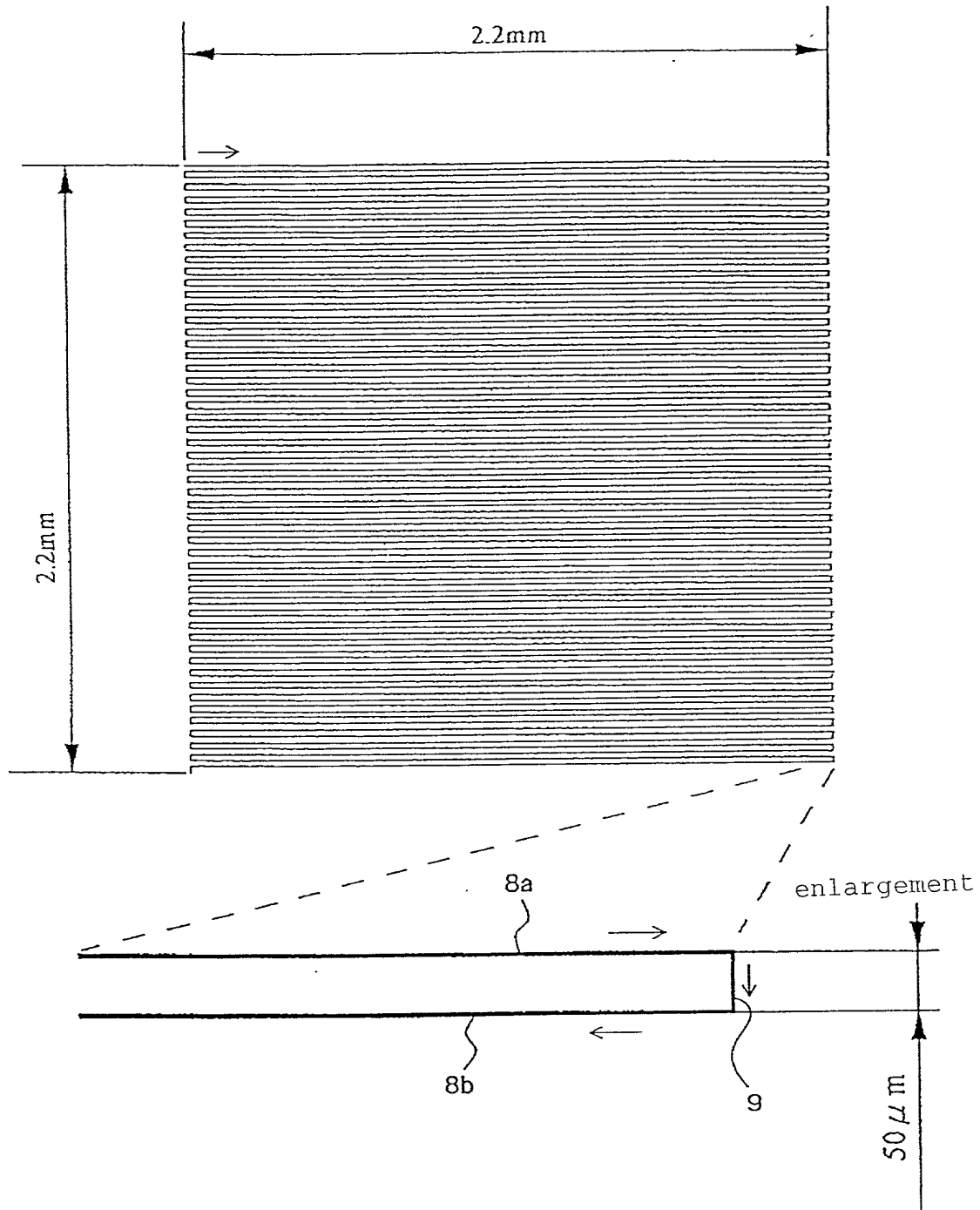


Fig. 8

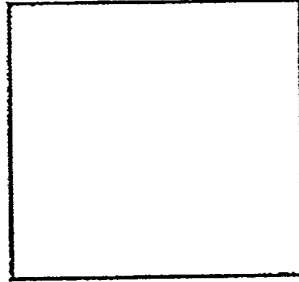


Fig. 9

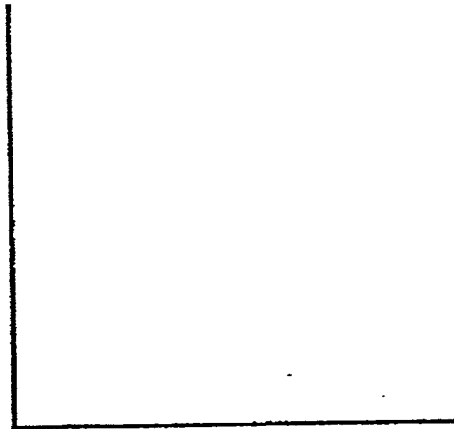


Fig. 10

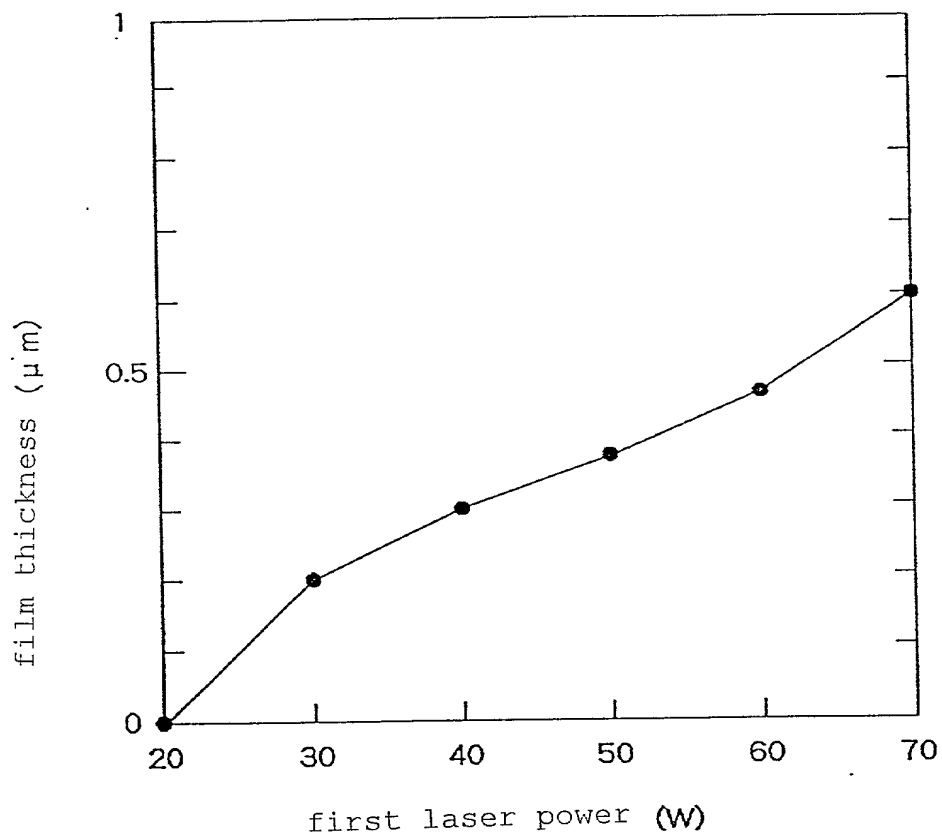


Fig. 11 (a)

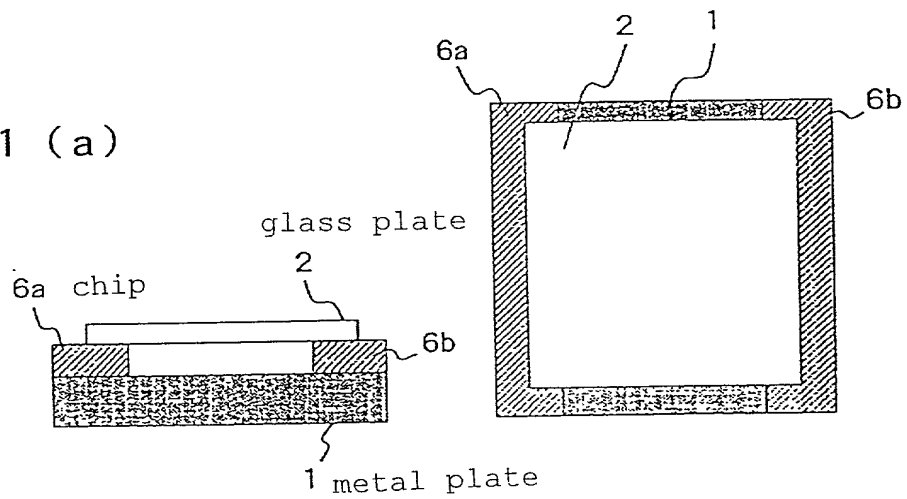


Fig. 11 (b)

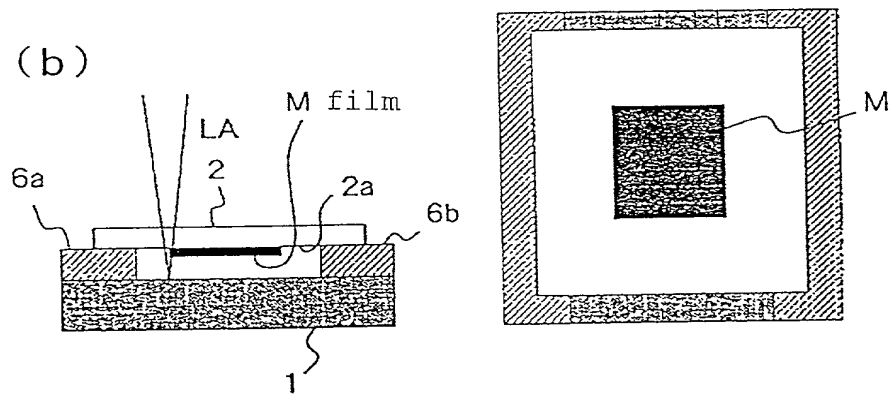


Fig. 11 (c)

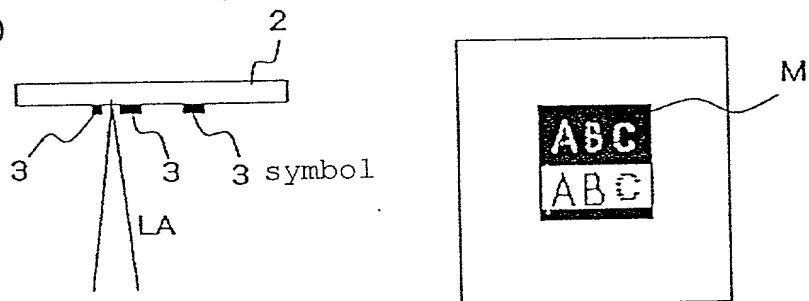


Fig. 12 (a)

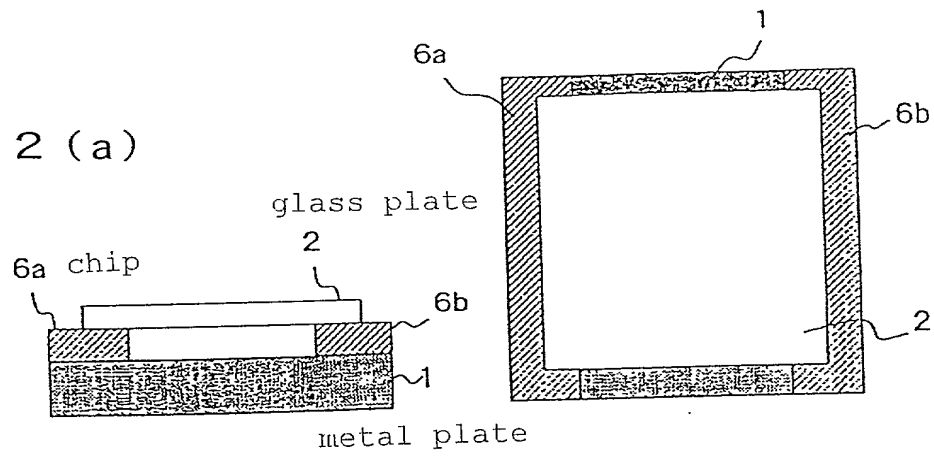


Fig. 12 (b)

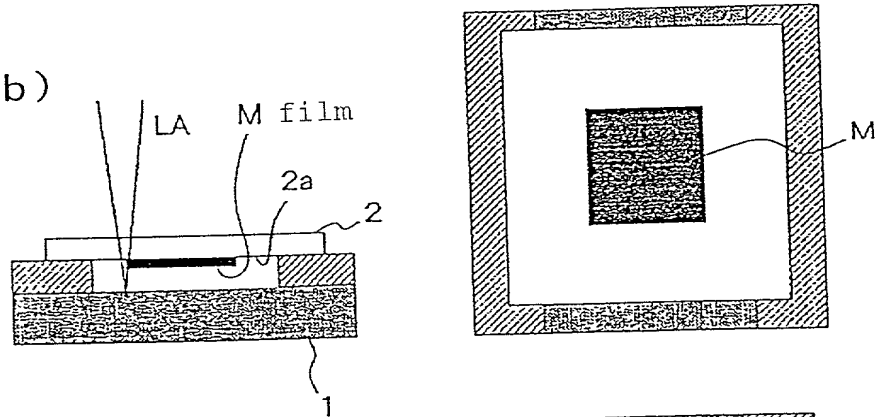


Fig. 12 (c)

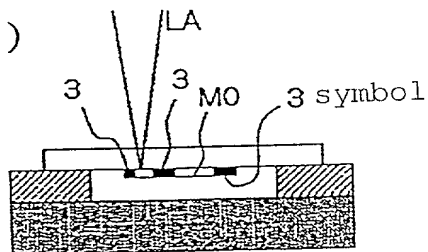


Fig. 12 (d)

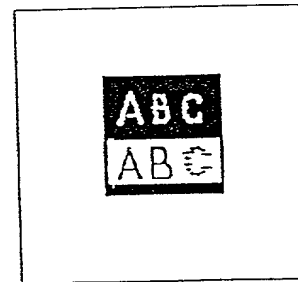


Fig. 13 (a)

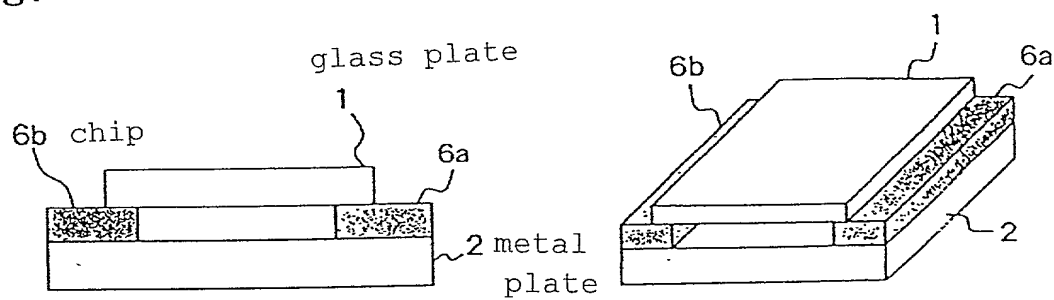


Fig. 13 (b)

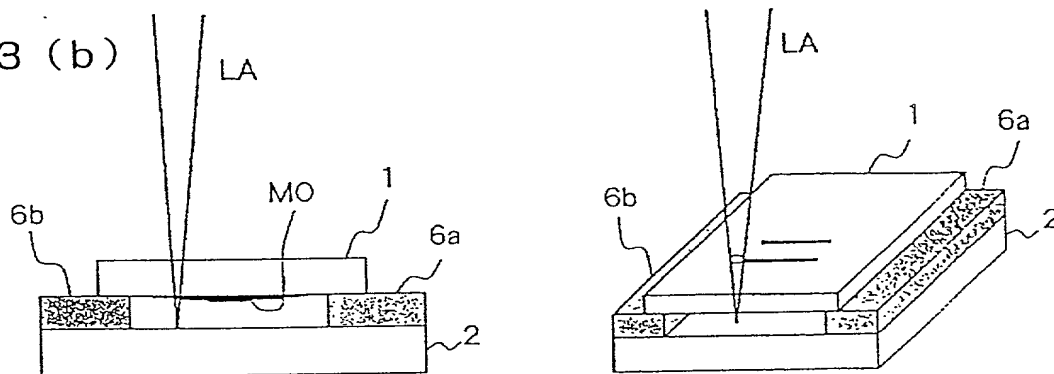


Fig. 13 (c)

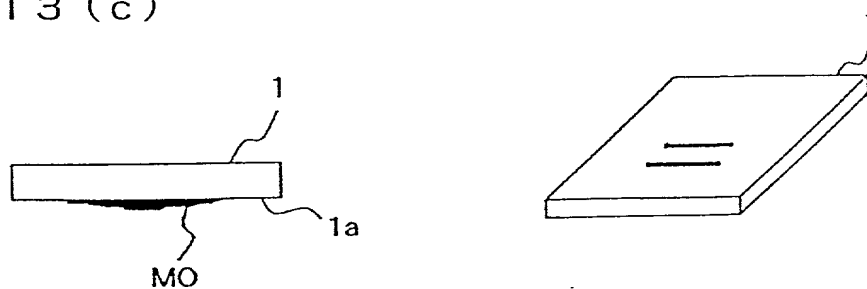


Fig. 14

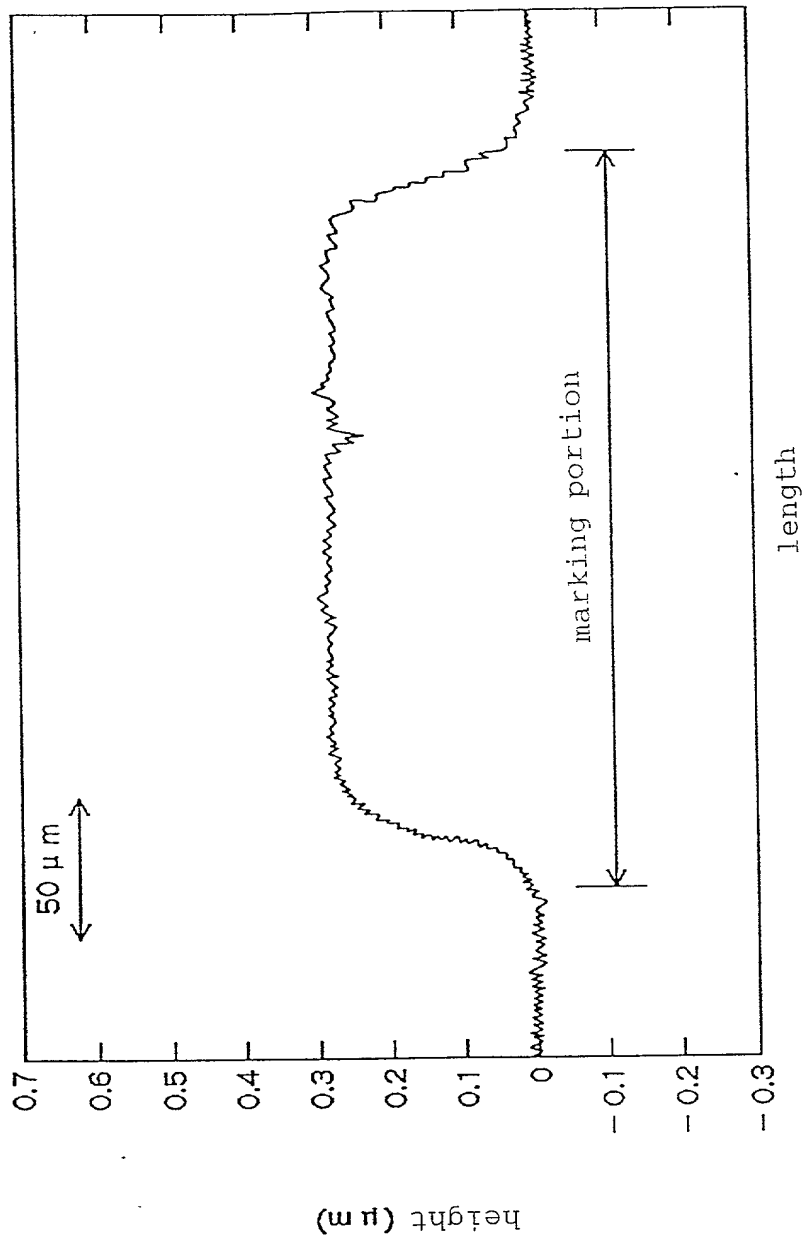


Fig. 15

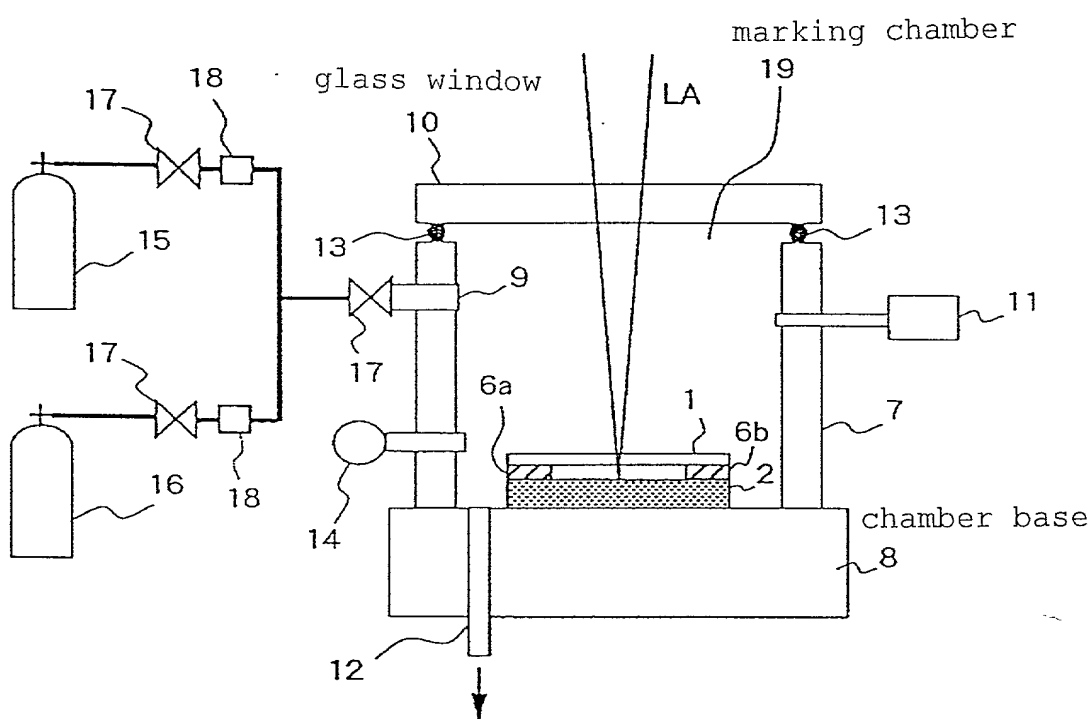
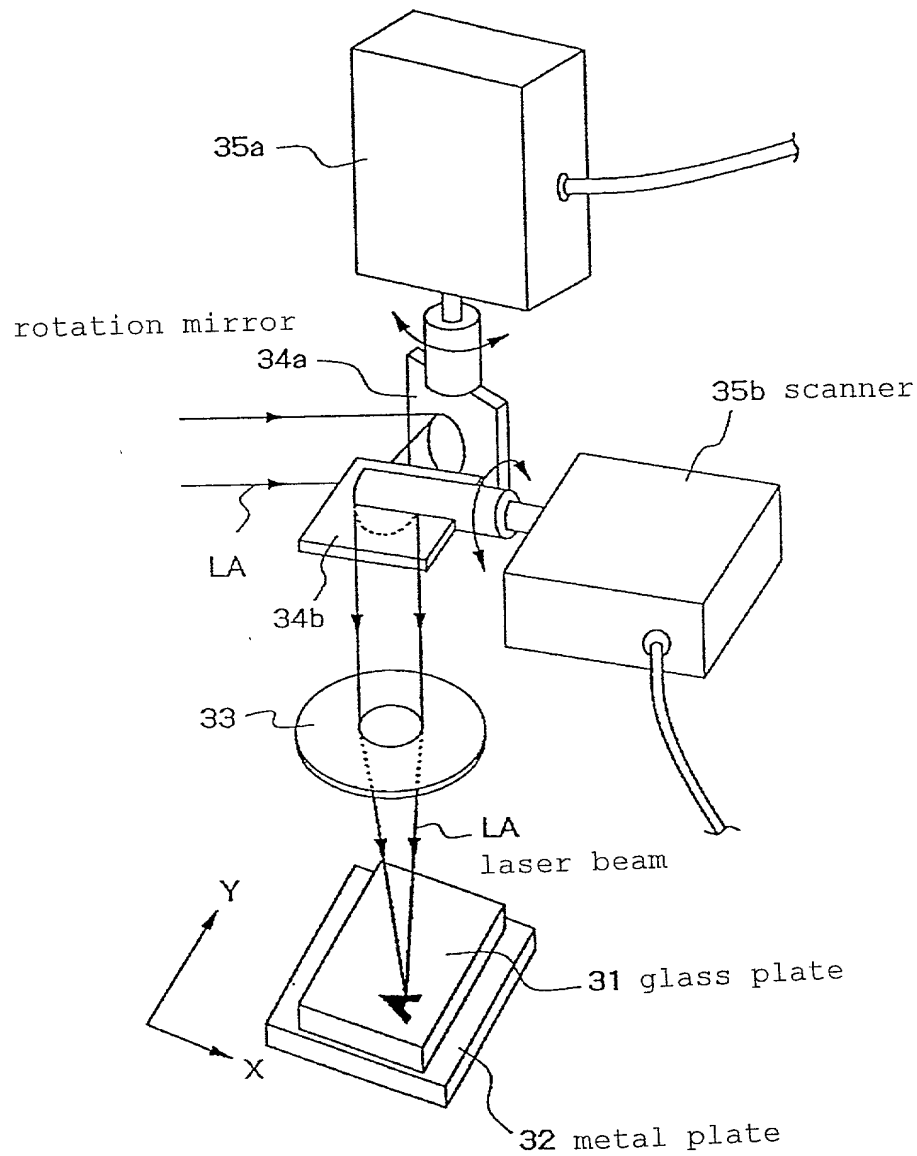


Fig. 16



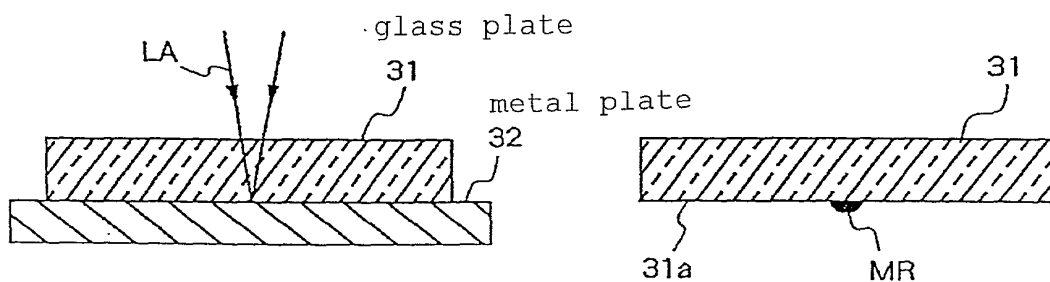
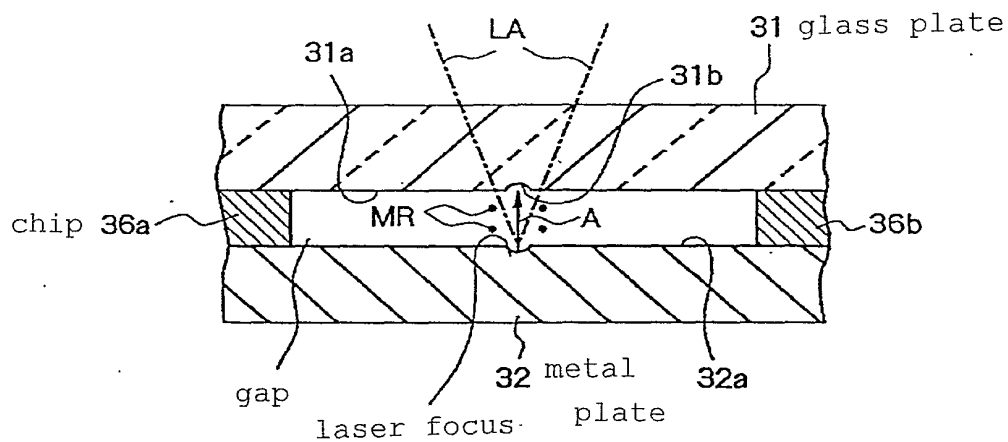


Fig. 17 (a)

Fig. 17 (b)

Fig. 18



Declaration and Power of Attorney For Patent Application

特許出願宣言書及び委任状

Japanese Language Declaration

日本語宣言書

下記の氏名の発明者として、私は以下の通り宣言します。

As a below named inventor, I hereby declare that:

私の住所、私書箱、国籍は下記の私の氏名の後に記載された通りです。

My residence, post office address and citizenship are as stated next to my name.

下記の名称の発明に関して請求範囲に記載され、特許出願している発明内容について、私が最初かつ唯一の発明者（下記の氏名が一つの場合）もしくは最初かつ共同発明者（下記の名称が複数の場合）であると信じています。

I believe I am the original, first and sole inventor (if only one name is listed below) or an original, first and joint inventor (if plural names are listed below) of the subject matter which is claimed and for which a patent is sought on the invention entitled.

METHOD FOR MARKING MATERIALS AND
MATERIALS USED FOR MARKING

上記発明の明細書は、

- ☐ 本書に添付されています。
- ☐ ____月____日に提出され、米国出願番号または特許協定条約国際出願番号を____とし、
(該当する場合) ____に訂正されました。

the specification of which

- ☐ is attached hereto.
- ☒ was filed on March 17, 1998
as United States Application Number or
PCT International Application Number
PCT/JP98/01110 and was amended on
____ (if applicable).

私は、特許請求範囲を含む上記訂正後の明細書を検討し、内容を理解していることをここに表明します。

I hereby state that I have reviewed and understand the contents of the above identified specification, including the claims, as amended by any amendment referred to above.

私は、連邦規則法典第37編第1条56項に定義されるとおり、特許資格の有無について重要な情報を開示する義務があることを認めます。

I acknowledge the duty to disclose information which is material to patentability as defined in Title 37, Code of Federal Regulations, Section 1.56.

Japanese Language Declaration

(日本語宣言書)

私は、米国法典第35編119条 (a) - (d) 項又は365条 (b) 項に基づき下記の、米国以外の国の少なくとも一カ国を指定している特許協力条約365 (a) 項に基づく国際出願、又は外国での特許出願もしくは発明者証の出願についての外国優先権をここに主張するとともに、優先権を主張している、本出願の前に出願された特許または発明者証の外国出願を以下に、枠内をマークすることで、示しています。

Prior Foreign Application(s)

外国での先行出願

9-068596

Japan

(Number)
(番号)

(Country)
(国名)

(Number)
(番号)

(Country)
(国名)

私は、第35編米国法典119条 (e) 項に基づいて下記の米国特許出願規定に記載された権利をここに主張いたします。

(Application No.)
(出願番号)

(Filing Date)
(出願日)

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(Application No.)
(出願番号)

(Filing Date)
(出願日)

(Application No.)
(出願番号)

(Filing Date)
(出願日)

私は、私自信の知識に基づいて本宣言書中で私が行なう表明が真実であり、かつ私の入手した情報と私の信じることに基づく表明が全て真実であると信じていること、さらに故意になされた虚偽の表明及びそれと同等の行為は米国法典第18編第1001条に基づき、罰金または拘禁、もしくはその両方により処罰されること、そしてそのような故意による虚偽の声明を行なえば、出願した、又は既に許可された特許の有効性が失われることを認識し、よってここに上記のごとく宣誓を致します。

I hereby claim foreign priority under Title 35, United States Code, Section 119 (a)-(d) or 365(b) of any foreign application(s) for patent or inventor's certificate, or Section 365(a) of any PCT International application which designated at least one country other than the United States, listed below and have also identified below, by checking the box, any foreign application for patent or inventor's certificate, or PCT International application having a filing date before that of the application on which priority is claimed.

Priority Claimed

優先権主張

☒ ☐
Yes No
はい いいえ
☐ ☐
Yes No
はい いいえ

21/March/1997

(Day/Month/Year Filed)
(出願年月日)

(Day/Month/Year Filed)
(出願年月日)

I hereby claim the benefit under Title 35, United States Code, Section 119(e) of any United States provisional application(s) listed below.

(Application No.)
(出願番号)

(Filing Date)
(出願日)

I hereby claim the benefit under Title 35, United States Code, Section 120 of any United States application(s), or Section 365(c) of any PCT International application designating the United States, listed below and, insofar as the subject matter of each of the claims of this application is not disclosed in the prior United States or PCT International application in the manner provided by the first paragraph of Title 35, United States Code Section 112, I acknowledge the duty to disclose information which is material to patentability as defined in Title 37, Code of Federal Regulations, Section 1.56 which became available between the filing date of the prior application and the national or PCT International filing date of application.

(Status: Patented, Pending, Abandoned)
(現況: 特許許可済、係属中、放棄済)

(Status: Patented, Pending, Abandoned)
(現況: 特許許可済、係属中、放棄済)

I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

Japanese Language Declaration
(日本語宣言書)

委任状：私は下記の発明者として、本出願に関する一切の手続きを米特許商標局に対して遂行する弁理士または代理人として、下記の者を指名いたします。
(弁理士、または代理人の指名及び登録番号を明記のこと)

POWER OF ATTORNEY: As a named inventor, I hereby appoint the following attorney(s) and/or agent(s) to prosecute this application and transact all business in the Patent and Trademark Office connected therewith: (list name and registration number)

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書類送付先

Send Correspondence to:

OBLON, SPIVAK, McCLELLAND, MAIER & NEUSTADT, P.C.
FOURTH FLOOR
1755 JEFFERSON DAVIS HIGHWAY
ARLINGTON, VIRGINIA 22202 U.S.A.

直接電話連絡先：(名前及び電話番号)

Direct Telephone Calls to: (name and telephone number)
(703) 413-3000

単独発明者または第一の共同発明者の氏名 1-00	Full name of sole or first joint inventor <u>Hirotooshi HAYAKAWA</u>
発明者の署名 日付	Inventor's signature <u>Hirotooshi Hayakawa</u> Date <u>August 3, 1999</u>
住所	Residence <u>Fukuoka, Japan JPX</u>
国籍	Citizenship <u>Japanese</u>
郵便の宛先	Post Office Address <u>c/o Kabushiki Kaisha Yaskawa</u> <u>Denki, 2-1, Kurosaki-Shiroishi,</u> <u>Yahatanishi-ku, Kitakyushu-shi,</u> <u>Fukuoka 806-0004 Japan</u>
第二の共同発明者の氏名 2-00	Full name of second joint inventor, if any <u>Shuichi TAKANAMI</u>
第二の共同発明者の署名 日付	Second joint Inventor's signature <u>Shuichi Takanami</u> Date <u>August 3, 1999</u>
住所	Residence <u>Saitama, Japan JPX</u>
国籍	Citizenship <u>Japanese</u>
郵便の宛先	Post Office Address <u>c/o YE DATA INC., 182, Oazashinko,</u> <u>Iruma-shi, Saitama 358-0055 Japan</u>

(第三以降の共同発明者についても同様に記載し、署名すること)

(Supply similar information and signature for third and subsequent joint inventors.)

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(21) 国際出願番号 PCT/JP98/01110 (22) 国際出願日 1998年3月17日(17.03.98) (30) 優先権データ 特願平9/68596 1997年3月21日(21.03.97) JP (71) 出願人 (米国を除くすべての指定国について) 株式会社 安川電機 (KABUSHIKI KAISHA YASKAWA DENKI)[JP/JP] 〒806-0004 福岡県北九州市八幡西区黒崎城石2番1号 Fukuoka, (JP) 株式会社 ワイ・イー・データ(YE DATA INC.)[JP/JP] 〒358-0055 埼玉県入間市大字新光182番地 Saitama, (JP) (72) 発明者; および (75) 発明者/出願人 (米国についてのみ) 早川博敏(HAYAKAWA, Hirotohi)[JP/JP] 〒806-0004 福岡県北九州市八幡西区黒崎城石2番1号 株式会社 安川電機内 Fukuoka, (JP) 高波修一(TAKANAMI, Shuichi)[JP/JP] 〒358-0055 埼玉県入間市大字新光182番地 株式会社 ワイ・イー・データ内 Saitama, (JP)	(74) 代理人 弁理士 若林 忠, 外(WAKABAYASHI, Tadashi et al.) 〒107-0052 東京都港区赤坂1丁目9番20号 第16興和ビル8階 Tokyo, (JP) (81) 指定国 CN, JP, KR, US, 欧州特許 (AT, BE, CH, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE). 添付公開書類 国際調査報告書	

(54) Title: **MARKING METHOD AND MARKING MATERIAL**

(54) 発明の名称 マーキング方法およびマーキング材

(57) Abstract

A marking method for forming a mark on the surface of an object to be marked by putting together the object of, e.g., a transparent body on which a pattern of characters, etc. are formed, and a marking material, and scanning the marking material with a laser beam, comprising a first step of irradiating the marking material with a laser beam of a first laser power and evaporating the marking material to form a deposit of the marking material on a predetermined portion of the object, and a second step of removing or modifying the deposit by irradiating the deposit with a laser beam of a second laser power. Steel or stainless steel is used as the marking material.

